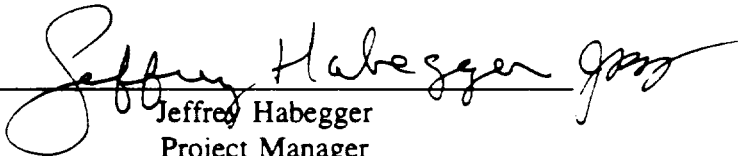


**FINAL CONTRACTOR'S SAMPLING AND ANALYSIS PLAN
FOR REMEDIATION OF LOCATIONS IN GRANITE CITY,
MADISON, AND VENICE, ILLINOIS, ASSOCIATED WITH
NL INDUSTRIES/TARACORP SUPERFUND SITE**

Submitted to:

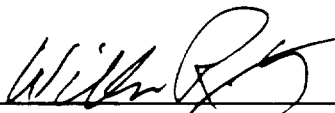
United States Army Corps of Engineers
Omaha, Nebraska

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November 29, 1993
Project 13407.3

TABLE OF CONTENTS

1.0	INTRODUCTION	1-1
1.1	SITE HISTORY	1-1
1.2	DESCRIPTION	1-2
1.3	PROJECT OBJECTIVES	1-2
1.4	FIELD ACTIVITIES	1-2
1.5	DISPOSAL SAMPLES	1-4
1.6	BACKFILL SAMPLING	1-4
1.7	CONTENTS	1-4
2.0	XRF SCREENING	2-1
2.1	AREA SCREENING	2-1
2.2	CONFIRMATION SCREENING	2-2
2.3	CONFIRMATION SAMPLING	2-2
2.4	ANALYTICAL SERVICES	2-2
3.0	CONFIRMATION SAMPLING	3-1
3.1	OBJECTIVES	3-1
3.2	TECHNICAL APPROACH	3-1
3.3	METHODOLOGY	3-1
3.4	DECONTAMINATION PROCEDURES	3-3
3.5	SAMPLE CONTROL	3-5
3.6	ANALYSIS	3-7
3.7	EQUIPMENT REQUIREMENTS	3-7
	FIGURE 3.1, OHM SHIPMENT CHECK LIST	3-4
4.0	EXCAVATED SOIL AND DECONTAMINATION LIQUID SAMPLING	4-1
4.1	OBJECTIVES	4-1
4.2	TECHNICAL APPROACH	4-1
4.3	SAMPLE COLLECTION FOR DISPOSAL ANALYSIS AND APPROVAL	4-1
4.4	SAMPLE COLLECTION FOR DISPOSAL DETERMINATION	4-6
4.5	SOIL SAMPLING METHODOLOGY	4-6
4.6	LIQUID SAMPLING METHODOLOGY	4-7
4.7	SAMPLE PACKAGING	4-9



TABLE OF CONTENTS (CONTINUED)

4.8	SAMPLE CONTROL	4-9
4.9	ANALYSIS	4-11
	TABLE 4.1, ANALYTICAL REQUIREMENTS LANDFILL DISPOSAL FOR SOLIDS AND SLUDGES	4-3
	TABLE 4.2, ANALYTICAL REQUIREMENTS DISPOSAL OF WASTEWATER LIQUIDS	4-12
	FIGURE 4.1, BACON BOMB SAMPLER	4-8
5.0	BACKFILL SAMPLING	5-1
5.1	OBJECTIVES	5-1
5.2	TECHNICAL APPROACH	5-1
5.3	METHODOLOGY	5-1
5.4	DECONTAMINATION PROCEDURES	5-3
5.5	SAMPLE CONTROL	5-4
5.6	ANALYSIS	5-6
5.7	EQUIPMENT REQUIREMENTS	5-6
6.0	ANALYTICAL REQUIREMENTS	6-1
6.1	METHODOLOGY	6-1
6.2	METHOD SPECIFIC DATA QUALITY OBJECTIVES	6-3
6.3	DELIVERABLES	6-6
	TABLE 6.1, SAMPLE TYPES AND NUMBERS	6-2
	TABLE 6.2, ANALYTICAL REQUIREMENTS FOR TREATED SOIL DISPOSAL	6-4
7.0	TREATED AND STOCKPILED SOIL SAMPLING	7-1
7.1	OBJECTIVES	7-1
7.2	TECHNICAL APPROACH	7-1
7.3	TREATED SOIL PILE SAMPLING METHODOLOGY	7-1
7.4	LIQUID SAMPLING METHODOLOGY	7-2
7.5	SAMPLE CONTROL	7-3
7.6	ANALYSIS	7-4
EXHIBIT I - PREVIOUS ANALYTICAL RESULTS PROVIDED BY WOODWARD-CLYDE CONSULTANTS		
EXHIBIT II - OPERATING MANUAL FOR THE SPECTRACE 9000 PORTABLE XRF ANALYZER		



TABLE OF CONTENTS (CONTINUED)

EXHIBIT III - STANDARD METHOD D-2216-80, MOISTURE CONTENT OF SOIL

EXHIBIT IV - STANDARD OPERATING PROCEDURES



1.0 INTRODUCTION

The United States Army Corps of Engineers (USACE) has tasked OHM Remediation Services Corp. (OHM), a wholly owned subsidiary of OHM Corporation, under the Rapid Response Contract No. DACW45-89-D-0516, Delivery Order No. 58, to perform soil removal activities associated with the NL Industries/Taracorp Superfund Site (NL Site) in Granite City, Illinois.

This Contractor's Sampling and Analysis Plan (CSAP) describes OHM's responsibilities with respect to the sampling and analysis associated with the work effort. OHM intends this document to be a site-specific guidance for the field team(s) for the project-required sampling and analysis.

1.1 SITE HISTORY

The NL Site is the location of a former secondary lead smelting facility. Prior to 1903, the facilities at the site included various smelting related equipment and processes. From 1903 to 1983, secondary lead smelting occurred on site. These activities were discontinued during 1983 and equipment dismantled.

In July of 1981, St. Louis Lead Recyclers, Inc. (SLLR) began using equipment on adjacent property owned by Trust 454 to separate components of the Taracorp waste pile. The objective was to recycle lead bearing materials to the furnaces at Taracorp and send hard rubber off site for recycling. SLLR continued operations until March 1983 when it shut down its equipment. Residuals from the operation remain on Trust 454 property as does some equipment.

A State Implementation Plan for Granite City, Illinois, was published in September 1983 by the Illinois Environmental Protection Agency (IEPA). The IEPA's report indicated that the lead nonattainment problem for air emissions in Granite City, Illinois, was in large part due to emissions associated with the operation of the secondary lead smelter operated by Taracorp and lead reclamation activities conducted by SLLR. The IEPA procured Administrative Orders by Consent with Taracorp, SLLR, Stackorp, Inc., Tri-City Truck Plaza, Inc., and Trust 454 during March 1984. The Orders required the implementation of remedial activities relative to air quality.

NL Industries (NL) as former owner of the site, voluntarily entered into an Agreement and Administrative Order by Consent with the United States Environmental Protection Agency (USEPA) and IEPA in May 1985 to implement a Remedial Investigation/Feasibility Study (RI/FS) for the site and other potentially affected areas. Taracorp was not a party to the agreement due to the fact that it filed for bankruptcy. The USEPA determined that the site was a Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) facility and it was placed on the National Priorities list on June 10, 1986.



1.2 DESCRIPTION

This action requires the excavation and disposal of fill material placed in alleys, parking lots, driveways, and yards in residential communities. The communities include Granite City, Madison, and Venice, Illinois. Based on the Record of Decision (ROD) the action levels established for this action will be 500 parts per million (ppm). Following the removal of the contaminated material, the areas impacted will be restored. This restoration will include sodding the yards, and paving the alleys, driveways, and parking lots.

1.3 PROJECT OBJECTIVES

The objectives of this field effort are to excavate any visible lead battery casings and slag and confirm that all contaminated soils have been removed to the action level of 500 ppm. Investigative sampling has been preformed by Woodward-Clyde Consultants (WOODWARD-CLYDE) Draft Final Report, NL/Taracorp Superfund Site, Granite City, Illinois, October 1992. Based on the initial study, the areas of excavation have been generally defined. OHM will follow the direction of USEPA's on-site representative to determine the limits of the excavation(s). OHM will incorporate X-Ray Fluorescence (XRF) screening to assist in complete removal of the contamination. After the material has been excavated, no more than five confirmatory samples will be collected. Samples of the excavated material will be obtained to determine appropriate disposal options.

The type of data needed to meet the project objectives will be generated through the installation of hand auger soil borings. The analytical methods required to meet the project objectives are total lead Methods 3050/7421 and TCLP RCRA metals Methods 1311/6010 and 7000 Series. A maximum of five confirmatory samples will be analyzed by total lead analysis. Two disposal samples will be analyzed by the TCLP RCRA metals analysis. One sample of the backfill will be analyzed for volatile and semivolatile organics and RCRA metals.

The cleanup criteria outlined in the ROD March 30, 1990, as defined by the USEPA requires the removal of all visual contamination from the alleyways and a cleanup criteria of 500 ppm of lead for the residential locations

1.4 FIELD ACTIVITIES

For all the locations, all visible battery casing contamination will be removed. A USEPA representative will be on site to assist in defining the removal area. At the residential areas, OHM will screen samples on site to quickly determine the levels of lead using XRF technology. The XRF screening will be performed to assist in removal of all material in the residential areas above 500 ppm lead. The XRF screening will be performed approximately every 20 feet in a staggered pattern to be representative of the area.

A maximum of five verification samples from the residential locations will be sent to an off-site laboratory for analysis. The areas will be backfilled and restored after visual/verification sampling.



The following are the field activities to be performed as part of the investigation.

1.4.1 Venice Alleys

Venice Alleys includes five alley areas that require removal of all contaminated material. It is anticipated that the material from the Klein and Lincoln Avenue alleys will be less than 5 milligrams per liter (mg/L) TCLP lead based on previous sampling. The material from Abbott Avenue, Slough Road, and Weber Avenue is expected to fail TCLP lead and may be segregated for disposal.

1.4.2 Eagle Park Areas

The Eagle Park areas require removal of all contaminated material. Personnel from USEPA will be present to determine the depth of the removal. In addition to the field screening data, one sample will be collected from the 203/205 Harrison location and one sample will be collected from the 203/205 Terry location for a total of two samples and analyzed for total lead. The material removed from the 108 Carver, 203/205 Harrison, 128 Roosevelt, and 208 Terry locations is expected to be below 5 ppm TCLP lead and may be segregated for disposal.

1.4.3 Missouri Avenue

Based on previous study, the excavation in the driveway will be to a depth of 1.5 to 2.0 feet. The southeast corner of the site will be excavated to a depth of 1 foot. One verification sample will be collected from this location in addition to the XRF field screening data. The material from this site is expected to fail TCLP lead and may be segregated for disposal.

1.4.4 2230 Cleveland

All contaminated material will be removed from the driveway/garage area at this site. Additional contamination is present in the yard, but is not part of this work effort. One verification sample will be collected from this location in addition to the XRF screening data. The material from this site is expected to fail TCLP lead and may be segregated for disposal.

1.4.5 1628 Delmar

The yard is overgrown at this location so it is difficult to define the extent of the visible contamination; therefore the entire yard will be screened. One verification sample will be collected from this location in addition to the XRF field screening data. The material removed is expected to be below 5 ppm TCLP lead and may be segregated for disposal.

1.4.6 3108 Colgate

The contamination in at this location appears to be limited to a fill area for the utilities on the east side of the residence. Because this area is residential, and the neighboring residents were uneasy with our presence during the site visit, it is important that OHM stay on the 3108 property



only for access. OHM will refer all residents questions to the USEPA or USACE. It is anticipated that the material from this site will fail TCLP lead and may be segregated for disposal.

1.5 DISPOSAL SAMPLES

Composite samples for disposal will be collected from the two stockpiles of contaminated soil and analyzed for disposal parameters. The purpose of two stockpiles is to separate soils that are expected to be more contaminated (based on the previous analytical from the investigative study) from the areas that are less contaminated. OHM will identify the disposal facility to be used for this work effort and the required disposal parameters for the facility. The expected analysis for disposal is TCLP RCRA metals. QA samples will not be required for these samples because the acceptance of the disposal facility validates the data.

Disposal samples will also be collected from the liquids generated as part of the proposed decontamination protocols. OHM anticipates the decontamination liquids will be collected and stored in a polyethylene aboveground storage tank. OHM will identify the disposal facility to be used for this work effort and the required disposal parameters for the facility. The expected analysis for disposal is TCLP RCRA metals. QA samples will not be required for these samples because the acceptance of the disposal facility validates the data.

1.6 BACKFILL SAMPLING

OHM will collect one sample of the backfill source to be used for the restoration activities outlined in the Draft Scope of Work, dated October 9, 1992. The anticipated analyses for the backfill sample include volatile and semivolatile organics, and RCRA metals.

1.7 CONTENTS

OHM intends for the activities described herein to comply and adhere to all applicable federal, state, and local laws and regulations and Applicable, Relevant, or Appropriate Requirements (ARARS), including the most recent USEPA guidelines as outlined in SW-846, and the USACE guidance document ER 1110-1-263 Appendix E.

This CSAP will include in the various sections that follow:

- ▶ A detail of all field and laboratory activities
- ▶ Documentation related to the chemical data
- ▶ A list of equipment to be taken to the field
- ▶ Details of the sampling locations and methodologies
- ▶ Field screening methods
- ▶ Decontamination procedures
- ▶ QC procedures
- ▶ Sample custody and shipment information
- ▶ Analytical methods



This CSAP is divided into individual sections. Each section is intended to be a standalone document related to the task described. Each individual section can be utilized by the field team(s) to accomplish the specific task. Additional detail, if needed, may be found in other sections or exhibits. The following topics are covered:

- ▶ Introduction
- ▶ XRF Screening
- ▶ Verification sampling
- ▶ Disposal/decontamination sampling
- ▶ Backfill sampling

Exhibits follow covering topics too detailed for inclusion in the body of this document and are for reference purposes.



2.0 XRF SCREENING

At each of the residential locations, OHM will determine remediation boundaries and monitor progress by screening for the target analyte, lead, with a portable XRF analyzer. OHM intends to use the Spectrace 9000 (TN Technologies, Inc., Round Rock, Texas) for XRF screening purposes. During all XRF operations, OHM personnel will record intensity and results data in the XRF instrument log. Please refer to Exhibit I, Operating Manual for the Spectrace 9000 Portable XRF Analyzer, for detailed operating procedures.

2.1 AREA SCREENING

To facilitate rapid determination of remediation boundaries, OHM will screen each residential area with the portable XRF analyzer before any remediation activities begin.

OHM will perform this screening by setting up the portable XRF analyzer in an office provided by USACE. The soil samples will be taken to the unit and analyzed. A microwave oven will be used to dry the samples to be screened. The soil samples will be placed into sample cups and these will be placed on the probe for analysis. The sample cups are made up of three pieces: cup, ring, and a piece of Mylar film. The cup will be filled nearly full with the soil sample to be screened. If the sample is powdered, the cup will be tapped to settle the contents and, if necessary, more sample added until the cup is at least 3/4 full. A piece of Mylar film will be approximately centered and placed over the open cup.

The ring will be placed over the cup with the rounded edge down. The ring will be pushed down slowly until it is flush with the end of the cup. The film will be taut and wrinkle-free. OHM understands that wrinkles cause part of the sample to be held away from the face of the probe and can interfere with the analysis. The filled and sealed sample cup will be inverted and tapped again to thoroughly settle and compact the contents. The sample will be placed film side down on the probe for analysis. The 30-millimeter ring will be placed in the large hole in the shield cup base to locate the sample cup reproducibly over the probe aperture. The OHM field chemist will ensure the sample cup rests in contact with the probe face. The probe button will be depressed and analysis of the sample will begin.

For each sampling location OHM personnel will record intensity and results data obtained from the XRF analyzer. Data from the XRF logs will be used to map the site remediation boundaries and target remediation activities to those areas above the action level of 500 mg/kg. The manufacturer reports that the detection limit for the Spectrace 9000 is typically 50 to 100 mg/kg for most analytes. Area screen soil samples will be acquired according to the procedures detailed in Section 3.3, Methodology.



2.2 CONFIRMATION SCREENING

Throughout the remediation activities, OHM will monitor progress by screening the excavation area with the portable XRF analyzer as previously described. OHM will screen with the portable XRF prior to each sampling event. Sampling locations will be randomly selected on each wall and on the floor of the excavation area. If screening data shows any section of the excavation to be below the lead action level (500 mg/kg), OHM will re-screen the entire area at randomly selected locations to determine remediation consistency. If levels are discovered to be greater than the action level, OHM will notify the OSR for further direction. OHM anticipates excavation to continue until all screening points are below the action level of 500 mg/kg. All XRF screening data, including intensity readings, will be recorded in the XRF instrument log.

2.3 CONFIRMATION SAMPLING

When XRF screening shows an excavated area to be below the action level (500 mg/kg) OHM will confirm this fact by sending several grab samples from each of the areas to be verified to EHRT Laboratory for total lead analysis. OHM will also obtain 10 percent field replicate samples from randomly selected residential excavations. EHRT Laboratory will be required to perform matrix quality control (QC) on one of OHM's QC samples. Please refer to Section 3.0, Confirmation Sampling for detail concerning the acquisition of the confirmation samples.

2.4 ANALYTICAL SERVICES

The name, address, and phone number of EHRT Laboratory is:

EHRT Laboratory
3532 Omni Drive
Cincinnati, OH 45245
513-752-2950
Contact: Dr. Mona Risk

OHM will use EHRT Laboratory to analyze the confirmation samples. OHM will expect EHRT Laboratory to follow in strict accordance to USEPA methods and to provide OHM with documentation which attests to this fact. EHRT Laboratory will be expected to provide OHM with a single-person contact for any discussions concerning OHM samples. This person will be expected to verify receipt, monitor progress, and appraise OHM of the status of its' samples. OHM requires immediate (24-hour) notification of laboratory problems with its' samples. A 24-hour turnaround time (from receipt of samples) will be requested for the confirmation samples.



3.0 CONFIRMATION SAMPLING

Five environmental soil samples, plus one duplicate, and one matrix spike/matrix spike duplicate will be collected to assess the residual contamination remaining after excavation and removal of the contaminated soils.

3.1 OBJECTIVES

This sampling effort is intended to assess the residual contamination remaining at the horizontal and vertical (lower) limits of the excavation. Systematic sampling will be used in combination with judgement sampling to acquire samples representative of the residual contamination remaining.

3.2 TECHNICAL APPROACH

OHM sample technologists will locate the post-excavation sample points after excavation activities are completed. XRF screening may be utilized to determine if verification samples will be taken and composited. Systematic screening techniques will be used to locate the sample points that will make up the single sample from the five areas to be sampled for verification analysis. The sample points to be composited will be located by using a systematic grid. Fence posts will be used to mark the horizontal limits of the excavation at the corners. The center grid point will be located horizontally by intersecting diagonals from these points and a grid will be superimposed on the area to be sampled in a similar manner as the screening sample points detailed in Section 2.0, XRF Screening. A sample will be acquired at each grid point, screened with the portable XRF, and combined with the other sample points from the same area for a single composite sample. Two duplicate samples will be chosen at random from two of the five selected areas for a total of seven field samples to be submitted for confirmation analysis at the contract laboratory.

3.3 METHODOLOGY

The OHM sample technologist will create a field sketch of the excavation area in a single plane. This sketch will be to approximate scale using the actual dimensions of the completed excavation. The center point will be located at the intersection of diagonal lines drawn from opposite corners of each of the four sidewall planes.

The sample technologist will thoroughly clean a stainless-steel tulip bulb planter (or equivalent) according to the protocols in Subsection 3.4, Decontamination, using soap/water/nitric acid. The sample points will be located horizontally on the surface to the nearest foot using a tape measure. Surveyor's stakes or flags will be used to locate the sample points at the surface. OHM anticipates very shallow excavations. If the excavations are determined to be unsafe to



enter based on depth, soil type, slope, and/or compaction, OHM will obtain samples by subsampling the bucket of the excavator. If the excavations are determined to be safe to enter the sample technologist will use the same techniques outlined below to acquire samples at the grid nodes.

The sample technologist will then dig down at these points until undisturbed soil at the sample location is retrieved. The sample technologist will don clean sample gloves and remove a clean stainless-steel spatula from its factory packaging or wrapping if decontaminated. The surface layer of soil will be scraped away with a disposable stainless-steel spatula. The sample technologist will once again don clean sample gloves and remove the clean hand auger from its wrapping. The clean hand auger will be inserted into the soil to its full depth and retrieved. These samples will be placed into a stainless-steel mixing bowl for compositing.

The sample technologist will once again don clean sample gloves and thoroughly mix the samples in the stainless-steel mixing bowl (Cole-Parmer Catalogue No. L-07300-50, or equivalent) to homogenize the contents. Large clods will be broken up if needed using a gloved hand until no piece is larger than 0.25 inches square. The stainless-steel spatula (VWR Catalogue No. 58575, or equivalent) will be used to thoroughly mix the contents of the bowl for no less than 3 minutes. The mixture will be spread into a thin layer and quartered. Opposite quarters will be discarded and the mixing, quartering, and splitting process continued until no less than two quarts by volume remains in the stainless steel bowl. This material will be transferred to each of one or two precleaned wide mouth clear glass 8-ounce sample jars with Teflon-lined lids (Eagle-Picher Catalogue No. 131-08C, or equivalent). The jars will be sealed and labeled. Clear tape will be placed over the label and custody seals will be applied to each container. Each sample will be placed into double Ziplock bags. The container will be wrapped in sorbent padding and placed into a sample cooler. A chain-of-custody record form will be completed and the sample will be documented in the field sampling notes. The stainless-steel utensils will be decontaminated again using the protocols detailed in Subsection 3.4, Decontamination. The sample will be shipped to the subcontract laboratory as detailed in Subsection 3.3.1, Sample Packaging, for Total Lead analysis. A 24-hour turnaround time will be requested (from time of sample receipt at the laboratory) in order to determine the residual contaminants remaining in the material in a timely manner.

The sampling items (used gloves, paper towels, disposable sampling gear, etc.) will then be placed in a disposal container. The samples will be labeled, packaged on ice, and their location documented on site sketches. This process will be repeated for each sample point location as well as the randomly selected duplicate sample points.

QA samples will be taken as described above. At two randomly selected areas, the composite sample for verification analysis will be split. One split will be submitted for duplicate analysis. The other randomly selected area composite sample will be submitted for matrix spike/matrix spike duplicate analysis by the selected laboratory. Each QC sample will be labeled and documented as such.



3.3.1 Sample Packaging

The samples will be wrapped with sorbent padding to reduce the chance of breakage in shipment and enclosed within a single plastic Ziplock bag. The bottom of the metal, or equivalent strength plastic shipping cooler will be lined with absorbent material, such as sorbent pads. The drain of the shipping container will be securely taped to prevent leakage in shipment. The wrapped containers will then be placed in the cooler allowing at least 1 inch of spacing between each container. Once the samples are secured, sorbent pads and ice will be placed on top of and among the sample containers. The remaining headspace in the cooler, if any, will then be filled with ice, followed by sorbent pads. Precautions will be taken to assure that the sample labels remain intact and legible.

The sample technologist will then sign the bottom of each chain-of-custody record form after Transfer Number 1. Under the heading "Transfers Relinquished By," he will fill in the courier's company name (such as Federal Express or UPS) and bill-of-lading number (or airbill number) as well as the date and time of sample custody relinquishment. The now completed chain-of-custody record forms will be enclosed in plastic Ziplock bags and taped to the underside of the lid of the cooler.

Prior to the sealing of the cooler, an OHM Shipment Checklist will be reviewed for completion. The checklist is a tool utilized by OHM to standardize sample packaging procedures during field operations. An example of the list is supplied (see Figure 3.1, OHM Shipment Check List).

A minimum of three custody seals or evidence tape will be fixed to the cooler lid(s). The cooler(s) will then be shipped to the subcontract laboratory via common courier. The cooler will be sealed, addressed, identified, and placarded as environmental samples. A 24-hour turnaround time from time of sample receipt at the laboratory will be requested.

3.4 DECONTAMINATION PROCEDURES

Decontamination of sampling equipment will be performed to ensure all of the contamination is removed in the contamination reduction zone before passing into the support zone.


All liquids and disposable clothing will be treated as contaminated waste and drummed or collected in bulk and staged accordingly. Personnel handling contaminated waste will wear Level C protection. All equipment leaving the exclusion zone will be cleaned prior to demobilization. Washwaters and residues will be drummed or bulked and staged accordingly.

3.4.1 Sampling Equipment

The field sampling equipment cleaning and decontamination procedures are as follows:

- ▶ Non-phosphate detergent wash and brushing to remove large particles



 OHM Corporation SHIPMENT CHECK LIST		
PROJECT NAME _____	PROJECT No.: _____	
STREET ADDRESS _____	DATE: / / TIME: _____	
CITY/STATE/ZIP _____	FAX NUMBER: () - _____	
PHONE NUMBER () - _____		

SAMPLE CHECK LIST			
	YES	NO	COMMENTS
SAMPLE LIDS ARE TIGHT AND CUSTODY SEALS IN PLACE?	<input type="checkbox"/>	<input type="checkbox"/>	
ARE ALL SAMPLE NUMBERS, DATES, TIMES AND OTHER LABEL INFORMATION LEGIBLE AND COMPLETE?	<input type="checkbox"/>	<input type="checkbox"/>	
HAS ALL SAMPLE NUMBERS, DATES, TIMES AND OTHER SAMPLING DATA BEEN LOGGED INTO THE SAMPLE LOG BOOK?	<input type="checkbox"/>	<input type="checkbox"/>	
DO SAMPLE NUMBERS AND SAMPLE DESCRIPTION ON THE LABELS MATCH WITH THOSE ON THE COC?	<input type="checkbox"/>	<input type="checkbox"/>	
HAVE THE SAMPLES BEEN PROPERLY PRESERVED?	<input type="checkbox"/>	<input type="checkbox"/>	
HAVE THE CHAIN OF CUSTODIES BEEN FILLED OUT COMPLETELY AND CORRECTLY?	<input type="checkbox"/>	<input type="checkbox"/>	
DOES THE ANALYTICAL SPECIFIED ON THE COC MATCH THE ANALYTICAL SPECIFIED IN THE SCOPE OF WORK?	<input type="checkbox"/>	<input type="checkbox"/>	
HAVE THE COC'S BEEN PROPERLY SIGNED IN THE TRANSFER SECTION?	<input type="checkbox"/>	<input type="checkbox"/>	

PACKAGING CHECK LIST			
	YES	NO	COMMENTS
HAS EACH SAMPLE BEEN PLACED INTO AN INDIVIDUAL PLASTIC BAG?	<input type="checkbox"/>	<input type="checkbox"/>	
HAS THE DRAIN PLUG OF THE COOLER BEEN TAPED CLOSED WITH WATER PROOF TAPE FROM THE INSIDE?	<input type="checkbox"/>	<input type="checkbox"/>	
HAS THE BOTTOM OF THE COOLER BEEN LINED WITH AT LEAST THREE INCHES OF CUSHIONING ABSORBANT PADS?	<input type="checkbox"/>	<input type="checkbox"/>	
HAVE ALL SAMPLES BEEN PLACED INTO THE COOLER IN AN UPRIGHT POSITION?	<input type="checkbox"/>	<input type="checkbox"/>	
IS THERE ADEQUATE SPACING OF SAMPLES SO THAT THEY WILL NOT TOUCH DURING SHIPMENT?	<input type="checkbox"/>	<input type="checkbox"/>	
HAVE AN ADEQUATE NUMBER OF BLUE ICE PACKS BEEN PLACED AROUND AND ON TOP OF THE SAMPLES?	<input type="checkbox"/>	<input type="checkbox"/>	
HAS FRESH BLUE ICE BEEN ADDED TO THE COOLER THE DAY OF SHIPMENT?	<input type="checkbox"/>	<input type="checkbox"/>	
HAS THE COOLER BEEN FILLED WITH ADDITIONAL CUSHIONING MATERIAL?	<input type="checkbox"/>	<input type="checkbox"/>	
HAS THE COC BEEN PLACED IN A ZIPLOCK BAG AND TAPED TO THE INSIDE OF THE LID OF THE COOLER?	<input type="checkbox"/>	<input type="checkbox"/>	
HAVE CUSTODY SEALS BEEN PLACED ONTO THE LID?	<input type="checkbox"/>	<input type="checkbox"/>	
HAS THE COOLER BEEN LABELED "THIS SIDE UP"?	<input type="checkbox"/>	<input type="checkbox"/>	
HAS THE COOLER BEEN LABELED WITH THE DOT PROPER SHIPPING NAME, UN/NA NUMBER AND PLACARD?	<input type="checkbox"/>	<input type="checkbox"/>	
HAS THE LABORATORY PERFORMING THE ANALYSES BEEN NOTIFIED OF THE SHIPMENT OF SAMPLES?	<input type="checkbox"/>	<input type="checkbox"/>	

PROBLEMS/RESOLUTIONS:	

PREPARED BY: _____	SIGNATURE: _____
--------------------	------------------



- ▶ Tap water rinse
- ▶ Sample glove change
- ▶ Ten percent nitric acid rinse (trace metal or higher grade HNO_3 diluted with distilled/deionized H_2O)
- ▶ Sample glove change
- ▶ Double distilled/deionized water rinse
- ▶ Total air dry

3.4.2 Sample Containers

Sample containers will be precleaned by the manufacturer to USEPA cleaning protocols prior to arrival at the project site as follows:

- ▶ All bottles, caps, and liners will be washed in laboratory grade, non-phosphate detergent
- ▶ These will be rinsed three times with distilled water
- ▶ Then they will be rinsed with a 1:1 solution of nitric acid
- ▶ They will then be rinsed three times with ASTM Type 1 organic-free water
- ▶ They will be oven-dried for 1 hour
- ▶ They will be rinsed with hexane
- ▶ Oven-dried again for 1 hour

3.5 SAMPLE CONTROL

Field personnel are responsible for the identification, preservation, packaging, handling, shipping, and storage of samples obtained from this site. All samples must be readily identifiable and retain to the extent possible the in-situ characteristics to be determined through testing. All samples collected to be analyzed for disposal parameters will be validated through the following procedures and preparations of a chain-of-custody record form.



3.5.1 Sample Containers

Soil samples taken for total lead analysis will be packaged in precleaned, 8-ounce wide-mouth, clear glass jars secured with a Teflon-lined lid (Eagle Picher No. 131-08C, or equivalent) pre-cleaned to USEPA Protocol A.

3.5.2 Sample Number

All samples obtained during the course of this project will be consecutively numbered. Each sample identification number includes a five-digit project code (OHM project number 13407) and a two-digit (or more as required) sequence number assigned by the sampler(s) at the time of collection. Post excavation samples will be consecutively numbered beginning with 10,001. The sample numbers will be recorded in consecutive order in a sample logbook. Field sketches will include the sample points and dimensions to trace the sample locations to the nearest foot within the excavations.

3.5.3 Sample Label

Correct sample labeling and the corresponding notation of the sample identification numbers in the field logbook are necessary to prevent misidentification of samples and their eventual results. All sample labels will be completed legibly with indelible ink. The completed label will be affixed to the sample bottle and covered with clear tape. All sample labels will include at a minimum the following information:

- ▶ Name/initials of collector
- ▶ Name/initials of witness
- ▶ Date and time (in military time) of sample collection
- ▶ OHM project number (13407)
- ▶ Place of collection
- ▶ Sample identification number (will uniquely identify each sample, i.e., project, station, location, depth interval, etc.)
- ▶ Matrix and appearance of sample
- ▶ Analysis required
- ▶ Preservatives added (if any)
- ▶ Designation between "grab" or "composite" sample



3.5.4 Field Log

OHM will record information from the sample collection activities in the sampler's field logbook. The log will be a diary of the sampler's activities and will contain sample point locations, appearance, date and time of sample, sampler's identity, and any other pertinent observations.

3.5.5 Chain-of-Custody Procedures

All samples taken on this site will be verified with chain-of-custody procedures. The procedures followed will be in accordance with USACE Sampling Handling Protocols and USACE procedures. It is very important that the information on the chain-of-custody record form match the information on the sample bottles. The chain-of-custody record forms will be completed, enclosed in a plastic Ziplock bag, and taped to the underside of the lid of the shipping containers utilized.

3.5.6 Sample Preservation

The post excavation soil samples submitted for confirmation analysis will be placed on ice to maintain each sample's temperature at 4 degrees Celsius.

3.6 ANALYSIS

The soil samples submitted for confirmation analysis will be analyzed according to USEPA's Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods, SW-846, 2nd Edition, September 1986. The soil samples submitted for Total Lead Analysis will be prepared by SW-846 Method 3050, "Acid Digestion of Sediments, Sludges, and Soils," followed by the Atomic Absorption Furnace Techniques detailed in Method 7421 for lead.

The samples for metals analysis will be analyzed within 6 months, the maximum allowable holding time. Soil and sediment sample results will be calculated and reported on a dry weight basis.

3.7 EQUIPMENT REQUIREMENTS

- ▶ Stainless-steel hand auger or equivalent (1)
- ▶ Stainless-steel mixing bowl (Cole-Parmer Catalogue No. L-07300-50, or equivalent) (1)
- ▶ Stainless-steel scoops (20)
- ▶ Stainless-steel spatulas (VWR Catalogue No. 58575, or equivalent) (200)
- ▶ Vinyl sample gloves (1,000)



- ▶ Non-phosphate detergent (14 ounces)
- ▶ Eight-ounce, wide-mouth, clear glass sample jars with Teflon-lined lids (Eagle-Picher No. 131-08C, or equivalent) precleaned to USEPA Protocol A (200)
- ▶ Fifty-four-quart coolers (5)
- ▶ Bale sorbent pads (1)
- ▶ Ice
- ▶ Paper towels
- ▶ Field logbook (1)
- ▶ Chain-of-custody record forms (150)
- ▶ Five-gallon buckets (4)
- ▶ 1:10 nitric acid (4 liters)
- ▶ Deionized water (10 liters)
- ▶ Scrub brush (1)
- ▶ Sample labels (300)



4.0 EXCAVATED SOIL AND DECONTAMINATION LIQUID SAMPLING

Excavation of the soils is necessary in the areas impacted from the battery casings. These soils are potentially contaminated and will need to be analyzed for treatment and disposal options to be considered.

The soils that are to be removed from the excavated areas will require sampling and analysis to define disposal/treatment options. The liquids generated as a result of decontamination activities will be collected and sampled for disposal/treatment options if they are not completely used for dust control measures.

This section of the CSAP deals with sampling and analysis of the soils and decontamination liquid generated from the excavation activities. All sampling procedures utilized will be in accordance with USACE Sample Handling Protocol (ER 1110-1-263, Appendix E).

4.1 OBJECTIVES

OHM will verify with treatment, storage, and disposal facilities, and define any additional analytical methods necessary for disposal of these wastestreams. OHM understands that all proposed protocols are subject to USACE approval and will inform the USACE-OSR of each step of the process.

4.2 TECHNICAL APPROACH

A flow based composite of the soils removed from each area will be obtained by subsampling selected buckets of the excavator during removal activities. Portable XRF screening may be used to screen and segregate soils removed for cost effectiveness of disposal activities. The soils excavated will be segregated based upon analytical data provided by WOODWARD-CLYDE included as Exhibit I to the project work plan.

OHM will combine liquids from the decontamination process into a portable bulk container staged at a suitable location (central site). A depth based composite of liquids generated as a result of decontamination activities will be obtained at the completion of this project as well. OHM anticipates that all decontamination liquids will be used for dust control measures and may not need to be sampled.

4.3 SAMPLE COLLECTION FOR DISPOSAL ANALYSIS AND APPROVAL

OHM intends to mobilize two personnel to the locations after delivery order issuance and prior to the mobilization of excavation equipment for the purposes of obtaining soil samples for



waste characterization and subcontractor acceptance/ approval(s). One composite of the hazardous areas and one composite of the nonhazardous areas will be created from no less than five grab samples from each respective wastestream. The location of the sample points will be based upon previous analytical results as detailed in Exhibit I to the work plan, Previous Analytical Results provided by WOODWARD-CLYDE. Judgment samples will be obtained at sample points within the areas delineated as hazardous and nonhazardous areas.

The sample technologist will don clean sample gloves and remove a stainless-steel spatula from its' factory packaging. The surface layer of soil in the selected bucket will be scraped away with a disposable stainless-steel spatula. The sample technologist will once again don clean sample gloves and remove a clean stainless-steel hand auger from its' wrapping. The clean hand auger will be inserted into the soil of the bucket to its' full depth (approximately 6 inches) and retrieved.

The sample technologist, using the hand auger, will place the soil obtained into a precleaned, 8-ounce, wide-mouth, clear glass sample jar (Eagle Picher No. 131-08C, or equivalent).

When the samples have been obtained and documented, the sample technologist will once again don clean sample gloves and thoroughly mix the various grab samples for disposal in the stainless-steel mixing bowl (Cole-Parmer Catalogue No. L-07300-50, or equivalent) to homogenize the contents. The grab samples representing the hazardous areas will be separate composites than those from the nonhazardous areas. Large clods will be broken up, if needed, using a gloved hand until no piece is larger than 0.25 inches square. The stainless-steel spatula (VWR Catalogue No. 58575, or equivalent) will be used to thoroughly mix the contents of the bowl for no less than 3 minutes or until the contents are homogenous. The mixture will be spread into a thin layer and quartered. Opposite quarters will be discarded and the mixing, quartering, and splitting process continued until no less than 2 quarts by volume remains in the stainless-steel bowl. This material will be transferred to each of four precleaned, 32-ounce, wide-mouth, clear glass sample jars with Teflon-lined lids (Eagle-Picher Catalogue No. 131-32C, or equivalent). The jars will be sealed and labeled. Clear tape will be placed over the sample label and a custody seal will be applied to each sample container to be shipped. Each sample container will be placed into two Ziplock bags. The containers will be wrapped in sorbent padding and placed into a sample cooler. The stainless-steel utensils will be decontaminated again using the protocols detailed in Section 3.4, Decontamination. One sample of each wastestream is for disposal analysis, the other three samples are for disposal acceptance purposes.

A chain-of-custody record form will be completed and one sample from each wastestream will be documented in the field sampling notes. One sample from each of the two wastestreams will be shipped to EHRT Laboratory in Cincinnati, Ohio, for Landfill Disposal analysis detailed in Table 4.1, the others will be shipped to the Analytical Services Corp., a wholly-owned subsidiary of OHM Corporation, (ASC) Laboratory in Findlay, Ohio, to be held prior to shipping them to the six potential disposal subcontractors. The six samples to be submitted to the various disposal subcontractors for disposal approvals will be sent with waste profiles upon receipt of the



TABLE 4.1
ANALYTICAL REQUIREMENTS
LANDFILL DISPOSAL FOR SOLIDS AND SLUDGES

Physical Tests:

Paint Filter Test, +/-
Color
Phasing
Solids, Total, % by Wt.
Density (bulk), g/cc

Characteristics Tests

<u>Reactivity</u>	<u>Method</u>	<u>LOD (mg/kg)</u>
Reactive Cyanide	9012	10
Reactive Sulfide	9030	10

Corrosivity, Standard Units

pH Test 9041

Ignitability, Degrees Fahrenheit or Celsius

Flash Point, SF, CC
Flame Test

Organics

Volatile Organics	8240	10/50
Semivolatile Organics	8270	10/50
PCB	8080	1.0

Conventional Tests

Extractable Organic Halogens	9075	1.0
Total Cyanide	9012	1.0
Total Phenols	9065	1.0



TABLE 4.1 (CONTINUED)

ANALYTICAL REQUIREMENTS
LANDFILL DISPOSAL FOR SOLIDS AND SLUDGES

Toxicity Leachate Characteristics Procedure (TCLP)

<u>Metals</u>	<u>Method LOD (ppm)</u>	<u>Reference</u>
Arsenic	0.1	7061
Barium	10.0	7081/6010
Cadmium	0.1	7130/7131/6010
Chromium	0.1	7190/7191/6010
Lead	0.1	7420/7421/6010
Mercury	0.01	7470
Selenium	0.1	7741
Silver	0.1	7760/7761/6010

Volatile Organics

Benzene	0.05	8260
Carbon tetrachloride	0.05	8260
Chlorobenzene	1.0	8260
Chloroform	0.5	8260
2-Butanone	5.0	8260
Tetrachloroethene	0.05	8260
Trichloroethene	0.05	8260
Vinyl chloride	0.05	8260

Semivolatiles

o-Cresol	5.0	8270
m-Cresol	5.0	8270
p-Cresol	5.0	8270
Cresol	5.0	8270
1,4-Dichlorobenene	0.5	8260/8270
1,2-Dichloroethane	0.05	8260
1,1-Dichloroethene	0.05	8260
2,4-Dinitrotoluene	0.01	8270
Hexachlorobenzene	0.01	8270
Hexachlorobutadiene	0.05	8270
Hexachloroethane	0.5	8270



TABLE 4.1 (CONTINUED)

ANALYTICAL REQUIREMENTS
LANDFILL DISPOSAL FOR SOLIDS AND SLUDGES

<u>Metals</u>	Method <u>LOD (ppm)</u>	<u>Reference</u>
<u>Semivolatiles</u>		
Nitrobenzene	0.1	8270
Pentachlorophenol	5.0	8270
Pyridine	0.5	8270
2,4,6-Trichlorophenol	0.1	8270



analytical results. A 21-day turnaround time will be requested (from time of sample receipt at the laboratory) for the disposal analysis of the nonhazardous soils. A 24-hour turnaround time (from time of sample receipt at the laboratory) will be requested for the disposal analysis of the hazardous soils.

4.4 SAMPLE COLLECTION FOR DISPOSAL DETERMINATION

The excavated soils will be placed into 1-cubic-yard, reinforced nylon bags (totes). These bags will be loaded into dumptrucks for transport to either a stockpile located in the Taracorp/Trust 454 property, if they originated in an area determined to be nonhazardous, based on the previous analytical results detailed in Exhibit I of the work plan, or transported directly to the selected hazardous-waste disposal subcontractor. The material determined to be hazardous based upon the previous analytical results will be transported directly to the chosen disposal subcontractor and will not be stockpiled. Samples of the excavated materials will be obtained and composited to confirm their hazardous/ nonhazardous properties by analysis for TCLP lead. Duplicate samples will be obtained on a 10 percent basis. The composite number representing each truckload of bags will be spray painted on two sides of each bag with numbers at least 1-foot high.

4.5 SOIL SAMPLING METHODOLOGY

A randomly selected grab sample from one bucketful of material of each tote will be taken and composited with the samples representing each dumptruck load (approximately 14 totes). These composites will be submitted for TCLP lead analysis to EHRT Laboratory. OHM anticipates approximately 205 composite samples for analysis.

To accomplish this task, the sample technologist will don clean sample gloves, remove a disposable stainless-steel spatula from its factory packaging, and scrape away the surface layer of soil in the selected bucket. Then the sample technologist will again don clean sample gloves, remove the clean hand auger or equivalent from its wrapping, and insert it into the soil of the bucket to its full depth (approximately 6 inches) and retrieve it.

The sample technologist will place the soil obtained into a precleaned 8-ounce, wide-mouth, clear glass sample jar (Eagle Picher No. 131-08C, or equivalent). These samples will eventually be composited into samples representing each truckload of totes.

When the excavation activities for the area are complete, the sample technologist will once again don clean sample gloves and thoroughly mix the various grab samples from the individual dumptruck loads in the stainless-steel mixing bowl (Cole-Parmer Catalogue No. L-07300-50, or equivalent) to homogenize the contents. Large clods will be broken up, if needed, using a gloved hand until no piece is larger than 0.25 square inches. The stainless-steel spatula (VWR Catalogue No. 58575, or equivalent) will be used to thoroughly mix the contents of the bowl for no less than 3 minutes or until the contents are homogenous. The mixture will then be spread into a thin layer and quartered. Opposite quarters will be discarded and the mixing, quartering, and splitting process continued until no less than 2 quarts by volume remains in the stainless-steel



bowl. This material will be transferred to one precleaned, 32-ounce, wide-mouth, clear glass sample jar with a Teflon-lined lid (Eagle-Picher Catalogue No. 131-32C, or equivalent). The jar will be sealed and labeled. Clear tape will be placed over the sample label, and a custody seal will be applied to each sample container to be shipped. Each sample container will be placed into two Ziplock bags, wrapped in sorbent padding, and placed into a sample cooler. The stainless-steel utensils will be decontaminated again using the protocols detailed in Section 3.4, Decontamination Procedures.

A chain-of-custody record form will be completed and the sample will be documented in the field sampling notes. The sample will be shipped to the subcontract laboratory for TCLP lead analysis.

4.6 LIQUID SAMPLING METHODOLOGY

OHM expects all decontamination water will be used for dust control during soil load-out operations. If decontamination water is not completely used, the remaining liquids will be sampled for disposal analysis.

The OHM sampling technician, clad in USEPA Level C personal protective equipment (PPE), will assemble a pre-cleaned, Teflon Bacon bomb sampler. The Bacon bomb sampler consists of a Teflon cylinder with a hole at the bottom and a weighted plunger to plug the hole (refer to Figure 4.1). The plunger protrudes through the top of the cylinder. There is a hole at the top of this rod where string can be attached to operate the plunger while sampling. The technician will attach a length of new sample string and a length of new 3/8-inch polyethylene rope to the Bacon bomb sampler. The rope will be marked in 1-foot increments or depths which will be needed for a representative sample of the tank. Polyethylene sheeting will be spread in the immediate vicinity of the sample point ensuring the sample string and rope do not touch any surface which may affect the integrity of the sample or the content of any tank. The Bacon bomb sampler will be lowered into the liquid(s) of the tank and an aliquot obtained at several depths by smoothly pulling on the string. This procedure will be repeated until the Bacon bomb sampler is full or the bottom of the tank is reached. Every attempt will be made to obtain equal aliquots at each depth. The Bacon bomb sampler will be retrieved and unloaded into a 1-liter glass sample container by placing it above the sample container and holding the plunger open with the attached string.

It is important that the surface of the liquid be visible to the sample technician at all times, because when the liquid flows into the sampling device, air is forced out of the top. The absence of air bubbles rising to the surface of the liquid lets the sample technician know when the Bacon bomb sampler is full.

The Bacon bomb sampler will be disassembled and thoroughly decontaminated prior to each use with a soap/water/nitric acid water rinse. The sample string and polyethylene rope used in the acquisition of samples will be discarded after each use.



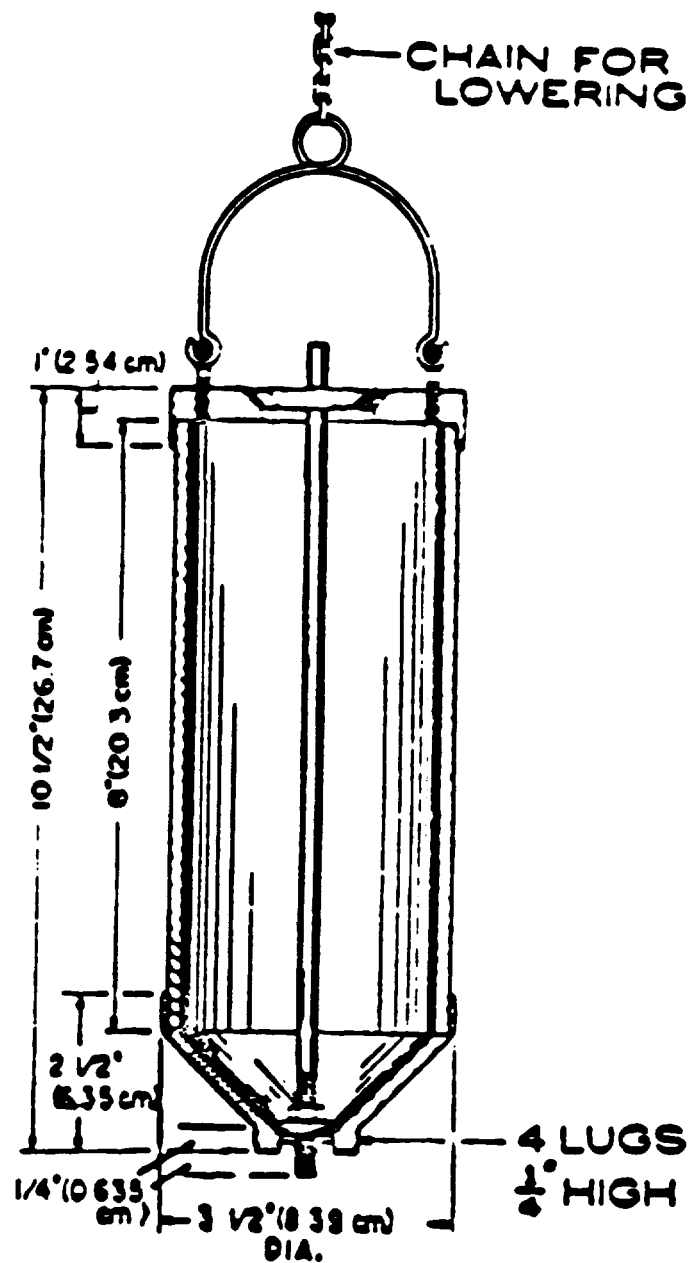


FIGURE 4.1
BACON BOMB SAMPLER



7.0 TREATED AND STOCKPILED SOIL SAMPLING

Soils that are taken to the NL Industries Site and treated to fix/stabilize the lead found in them will be temporarily stockpiled and will need to be sampled and analyzed.

This section of the CSAP deals with sampling and analysis of the treated stockpiled soil materials that resulted from the excavation activities. All sampling procedures utilized will be in accordance with USACE Sample Handling Protocol (ER 1110-1-263, Appendix E).

7.1 OBJECTIVES

OHM will verify with treatment, storage, and disposal facilities and define any additional analytical methods necessary for disposal of this wastestream. OHM understands that all proposed protocols are subject to USACE approval and will inform the USACE-OSR of each step of the process. The objectives of this section are as follows:

- ▶ Obtain representative samples of the treated soils
- ▶ Describe the methods to be used to collect samples

7.2 TECHNICAL APPROACH

OHM sample technicians will obtain one sample of treated soil for approximately every 100 cubic yards of treated material. This material will be obtained immediately after it has come off the pugmill exit conveyor. The material will be sent by overnight courier service to the subcontract laboratory and then be analyzed.

OHM will combine liquids from the decontamination process into a portable bulk container staged at a suitable location (central site). A depth based composite of liquids generated as a result of decontamination activities will be obtained at the completion of this project as well.

7.3 TREATED SOIL PILE SAMPLING METHODOLOGY

Treated soil pile samples will be obtained by the procedures described in OHM Standard Operating Procedure QP-613 "Pile Sampling" (see Exhibit IV). As long as the piles are still "wet" and have not set up, the procedures will work. If the pile has set up, a hammer and chisel approach to obtaining a sample will be required.

A chain-of-custody record form will be completed and the sample will be documented in the field sampling notes. The sample will be sent to the subcontract laboratory for analysis. A minimum turnaround time will be required from the time of sample receipt at the laboratory until results are available.



7.4 LIQUID SAMPLING METHODOLOGY

The OHM sampling technician, clad in USEPA Level C PPE, will assemble a pre-cleaned, Teflon Bacon bomb sampler. The Bacon bomb sampler consists of a Teflon cylinder with a hole at the bottom and a weighted plunger to plug the hole. The plunger protrudes through the top of the cylinder. There is a hole at the top of this rod where string can be attached to operate the plunger while sampling. The technician will attach a length of new sample string and a length of new 3/8-inch polyethylene rope to the Bacon bomb sampler. The rope will be marked in 1-foot increments or depths which will be needed for a representative sample of the tank. Polyethylene sheeting will be spread in the immediate vicinity of the sample point ensuring the sample string and rope do not touch any surface which may affect the integrity of the sample or the content of any tank. The Bacon bomb sampler will be lowered into the liquid(s) of the tank and an aliquot obtained at several depths by smoothly pulling on the string. This procedure will be repeated until the Bacon bomb sampler is full or the bottom of the tank is reached. Every attempt will be made to obtain equal aliquots at each depth. The Bacon bomb sampler will be retrieved and unloaded into a 1-liter glass sample container by placing it above the sample container and holding the plunger open with the attached string.

It is important that the surface of the liquid be visible to the sample technician at all times, because when the liquid flows into the sampling device, air is forced out of the top. The absence of air bubbles rising to the surface of the liquid lets the sample technician know when the Bacon bomb sampler is full.

If the Bacon bomb sampler is filled before the bottom of the tank is reached, the depth will be noted and the device retrieved, unloaded, and reinserted into the tank to the deepest depth noted and the procedure repeated as previously mentioned.

The Bacon bomb sampler will be disassembled and thoroughly decontaminated prior to each use with a Alconox/isopropanol/deionized water rinse. The sample string and polyethylene rope used in the acquisition of samples will be discarded after each use.

7.4.1 Sample Packaging

The samples will be wrapped with sorbent padding to reduce the chance of breakage in shipment and enclosed within a single plastic Ziplock bag. The bottom of the metal, or equivalent strength, plastic shipping cooler will be lined with absorbent material, such as sorbant pads. The drain of the shipping container will be securely taped to prevent leakage in shipment. The wrapped containers will then be placed in the cooler allowing at least 1 inch of spacing between each container. Once the samples are secured, sorbent pads and ice will be placed on top of and among the sample containers. The remaining headspace in the cooler, if any, will then be filled with ice followed by additional bubble pack. Precautions will be taken to assure that the sample labels remain intact and legible.



The sample technologist will then sign the bottom of each chain-of-custody record form after Transfer No. 1 under the heading "Transfers Relinquished By," and indicate the courier's company name (such as Federal Express or UPS), the bill-of-lading number (or airbill number), and the date and time of sample custody relinquishment. The now-completed chain-of-custody record forms will be enclosed in plastic Ziplock bags and taped to the underside of the lid of the cooler.

Prior to the sealing of the cooler, an OHM Shipment Check List will be reviewed for completion. The check list is a tool utilized by OHM to standardize sample packaging procedures during field operations.

A minimum of three custody seals or evidence tape will be fixed to the cooler lid(s). The cooler(s) will then be shipped to the subcontract laboratory via common courier. The cooler will be sealed, addressed, identified, and placarded as environmental samples. A 48 hour or less turnaround time will be requested (from time of sample receipt at the laboratory).

7.5 SAMPLE CONTROL

OHM field personnel are responsible for the identification, preservation, packaging, handling, shipping, and storage of samples obtained from this site. All samples will be readily identifiable and retain to the extent possible the in-situ characteristics to be determined through testing. All samples collected to be analyzed by a laboratory will be validated through the following procedures and preparation of a chain-of-custody record form.

7.5.1 Sample Number

All samples obtained during the course of this project will be consecutively numbered. Each sample identification number includes a five-digit project code (OHM project number) and a two-digit (or more as required) sequence number assigned by the sampler(s) at the time of collection. The sample numbers will be recorded in consecutive order in a sample logbook.

The sample numbers to be used for treated soil samples will be consecutively numbered beginning with 70,001. Liquid samples for disposal analyses will remain the same as in Section 4.6.1 as will the liquid samples submitted to selected vendors for disposal acceptance.

7.5.2 Sample Label

Correct sample labeling and the corresponding notation of the sample identification numbers in the field logbook are necessary to prevent misidentification of samples and their eventual results. All sample labels will be completed legibly with indelible ink. The completed label will be affixed to the sample bottle and covered with clear tape. All sample labels will include at a minimum the following information:

- ▶ Name/initials of collector



- ▶ Name/initials of witness
- ▶ Date and time (in military time) of sample collection
- ▶ OHM project number
- ▶ Place of collection
- ▶ Sample identification number (will uniquely identify each sample, i.e., project, station location, depth interval, etc.)
- ▶ Matrix and appearance of sample
- ▶ Analysis required
- ▶ Preservatives added (if any)
- ▶ Designation between "grab" or "composite" sample

7.5.3 Field Log

OHM will record information from the sample collection activities in the sampler's field logbook. The log will be a diary of the sampler's activities and will contain sample point locations, appearance, date and time of sample, sampler's identity, and any other pertinent observations.

7.5.4 Chain-of-Custody Procedures

All samples taken on this site will be verified with chain-of-custody procedures. The procedures followed will be in accordance with USACE Sampling Handling Protocols and USACE procedures. It is very important that the information on the chain-of-custody record form match the information on the sample bottles. The chain-of-custody record forms will be completed, enclosed in a plastic Ziplock bag, and taped to the underside of the lid of the shipping containers utilized.

7.5.5 Sample Preservation

The post treatment soil samples submitted for analysis to the subcontract laboratory will be held/stored at room temperature so that curing of the material can occur.

7.6 ANALYSIS

The soil samples will be analyzed in accordance with USEPA Method 1311 and in accordance with Method 7420. For disposal purposes, a special set of analytical parameters is given in Table 6.2.



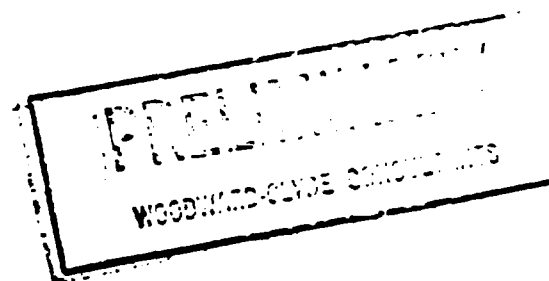
The water samples submitted for disposal parameter analysis will be analyzed according to USEPA's Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update I, September 1986.



EXHIBIT I

**PREVIOUS ANALYTICAL RESULTS PROVIDED BY
WOODWARD-CLYDE CONSULTANTS**

TABLE 8



**SAMPLE IDENTIFICATION CODE DESCRIPTION
NL/TARACORP SUPERFUND SITE**

Each sample has a unique sample identification. The identification consists of sample matrix code, street code, lot number, boring number, sample depth code, and sample type. All of the codes are listed in the following tables with their appropriate description. An example follows to demonstrate the operation of the sample identification.

SMP1629200B00L

- S** Sample Matrix (In this case, the sample matrix is soil, see **SAMPLE MATRICES** table.)
- MP** Street Code (In this case, the sample location is on Maple Street, see **STREET CODE** table.)
- 1629** Lot Number (In this case, the sample was taken at lot/house number 1629.)
- 2** Boring Number (In this case, the sample was taken from the 2nd boring on the property.)
- 00B** Sample Depth (In this case, the sample was taken between 3 - 6 inches from the boring indicated, see **SAMPLE DEPTHS** table.)
- 00L** Sample Type (In this case, the sample was analyzed for Total Lead, see **SAMPLE TYPES** table.)

SAMPLE MATRICES

- S** Soil Sampled for Chemical Analysis &/or Geotechnical
- W** Groundwater Sampled from Monitoring Wells

TABLE 8

SAMPLE IDENTIFICATION CODE DESCRIPTION
NL/TARACORP SUPERFUND SITE

STREET CODES

RESIDENTIAL

AD	ADAMS
AL	ALTON
BE	BENTON
BR	BRYAN
CH	CHESTNUT
CL	CLEVELAND
DE	DELMAR
DV	DENVER
ED	EDISON
EL	ELIZABETH
ER	EDWARDSVILLE ROAD
GR	GRAND
GW	GREENWOOD
IO	IOWA
KE	KENNEDY
LE	LEE
MA	MADISON
MP	MAPLE
MC	McCAMBRIDGE
ME	MEREDOCIA
NI	NIEDRINGHAUS

OH	OHIO
OL	OLIVE
RE	REYNOLDS
RR	ROCK ROAD
SA	SALVETER
SP	SPRUCE
ST	STATE
WA	WALNUT
WS	WASHINGTON

ET	18th
FI	5th
NT	19th
TL	12th
SN	2nd
TW	20th
TS	22nd
WT	W. 20th

INDUSTRIAL AND REMOTE FILL AREAS

BV	BV & G TRANSPORT
OR	OTHER REMOTE FILL AREAS
RO	RICH OIL
TA	TARACORP
TR	TRUST 454
VE	VENICE ALLEYS

CA	CARVER
CO	COLGATE
HA	HARRISON
HI	HILL
RS	ROOSEVELT
TE	TERRY

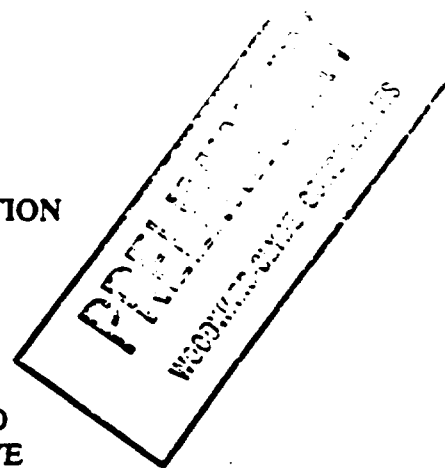


TABLE 8
SAMPLE IDENTIFICATION CODE DESCRIPTION
NL/TARACORP SUPERFUND SITE

SAMPLE DEPTH	
<u>CODE</u>	<u>DEPTH</u>
00A	0-3 inches
00B	3-6 inches
00C	6-12 inches
00D	1-2 feet
00E	2-3 feet
00F	3-4 feet
00G	4-5 feet
00H	5-6 feet
00I	6-7 feet
00J	0-2 feet
00K	2-4 feet
00L	4-6 feet
00M	6-8 feet
00N	8-10 feet
00P	10-12 feet
00R	12-14 feet
00S	14-15 feet
00T	13-15 feet
00U	10-11 feet
00V	15-16 feet
00W	20-21 feet
00X	25-26 feet
0AB	0-6 inches
0AC	0-1 feet
0GG	Top of Groundwater

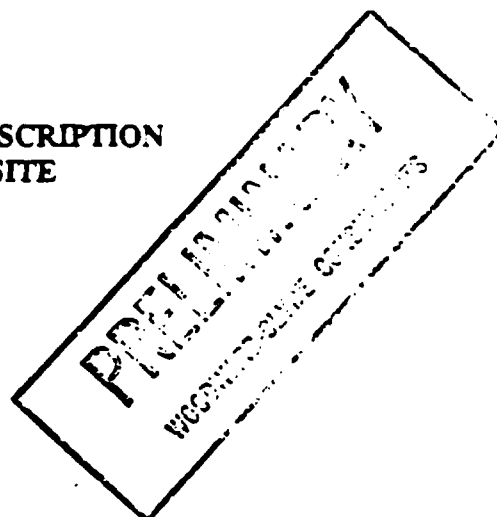
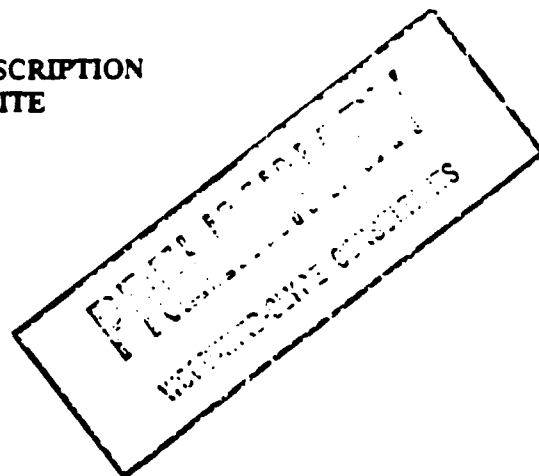


TABLE 8

**SAMPLE IDENTIFICATION CODE DESCRIPTION
NL/TARACORP SUPERFUND SITE**

SAMPLE TYPE

00G	Geotechnical Sample
0GD	Geotechnical Duplicate
0GQ	Geotechnical QA Sample
00L	Total Lead Sample
0LD	Total Lead Duplicate Sample - Boring 1
0LQ	Total Lead Quality Assurance
0XM	Total Lead, Boring 2, Duplicate - # 1
0XX	Total Lead, Boring 2, Duplicate - # 2
00T	TCLP Lead Sample
0TD	TCLP Lead Duplicate
0TQ	TCLP Lead Quality Assurance
0TM	TCLP Lead Matrix Spike
0TX	TCLP Lead Matrix Spike Duplicate
00W	Groundwater Sample
0WD	Groundwater Duplicate
0WB	Groundwater Rinsate Blank
0WM	Groundwater Matrix Spike
0WX	Groundwater Matrix Spike Duplicate
0WQ	Groundwater QA Sample
0WR	Groundwater QA Matrix Spike
0WS	Groundwater QA Matrix Spike Duplicate
0WT	Groundwater QA Rinsate Blank
0TB	Trip Blank
0RS	Re-Sample



CA - Carbon
1 - Total Lead

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**TABLE 24
EAGLE PARK DATA SUMMARY**

NL/TARACORP 89MANALYTICAL REPORT

GENERATED: Sep 02, 1992

SAMPLE ID	PARAMETER	SAMPLE COLLECTION DATE	ANALYSIS DATE	RESULT	QUALIFIER	UNITS	REPORTING DETECTION LIMIT	PARAMETER	ANALYSIS DATE	RESULT	UNITS
SCA0108100C00L	Total Lead	05/19/1992	07/29/1992	154	J	MG/KG	5.1	Moisture Content	07/08/1992	19.7	%WET W
SCA0108100A00T	TCLP Lead	05/19/1992		4		MG/L					
SCA0108200C00L	Total Lead	05/19/1992	07/29/1992	1810	J	MG/KG	5.3	Moisture Content	07/08/1992	9.9	%WET W
SCA010820AB00L	Total Lead	05/19/1992	07/29/1992	4350	J	MG/KG	29.0	Moisture Content	07/08/1992	14.5	%WET W
SCA011110AB00L	Total Lead	05/19/1992	07/29/1992	471	J	MG/KG	5.4	Moisture Content	07/08/1992	9.1	%WET W
SCA011120AB00L	Total Lead	05/19/1992	07/29/1992	445	J	MG/KG	5.2	Moisture Content	07/08/1992	9.2	%WET W
SHA0202100C00L	Total Lead	05/27/1992	07/30/1992	2320		MG/KG	5.1	Moisture Content	07/10/1992	23.3	%WET W
SHA0202100C00T	TCLP Lead	05/27/1992		11.7		MG/L	0.2				
SHA0202100D00L	Total Lead	05/27/1992	07/30/1992	103		MG/KG	5.9	Moisture Content	07/10/1992	22.1	%WET W
SHA0202100E00L	Total Lead	05/27/1992	07/30/1992	198		MG/KG	5.0	Moisture Content	07/10/1992	21.1	%WET W
SHA020210AB00L	Total Lead	05/27/1992	07/30/1992	68400		MG/KG	261	Moisture Content	07/10/1992	10.2	%WET W
SHA020210AB00T	TCLP Lead	05/27/1992	09/15/1992	440		MG/L	0.18				
SHA0202200C00T	TCLP Lead	05/27/1992	09/15/1992	2.34		MG/L	0.18				
SHA0202200F00L	Total Lead	05/27/1992	07/30/1992	19.4		MG/KG	5.1	Moisture Content	07/10/1992	22.9	%WET W
SHA020220AB00L	Total Lead	05/27/1992	07/30/1992	1240		MG/KG	5.1	Moisture Content	07/10/1992	10.0	%WET W
SHA020220AB00T	TCLP Lead	05/27/1992	09/15/1992	1.47		MG/L	0.18				
SHA0202300C00L	Total Lead	05/27/1992	07/30/1992	752		MG/KG	5.1	Moisture Content	07/10/1992	22.7	%WET W
SHA0202300E00L	Total Lead	05/27/1992	07/30/1992	622		MG/KG	5.6	Moisture Content	07/10/1992	28.3	%WET W
SHA0202300F00T	TCLP Lead	05/27/1992	09/15/1992	0.93		MG/L	0.18				
SHA0202300F0TD	TCLP Lead	05/27/1992	09/15/1992	1.13		MG/L	0.18				
SHA0202300G00L	Total Lead	05/27/1992	07/30/1992	177		MG/KG	5.9	Moisture Content	07/10/1992	28.3	%WET W
SHA020230AB0L	Total Lead	05/27/1992	07/30/1992	937		MG/KG	5.5	Moisture Content	07/10/1992	9.1	%WET W
SHA020230AB0LD	Total Lead	05/27/1992	07/30/1992	536		MG/KG	5.3	Moisture Content	07/10/1992	11.6	%WET W
SHA0202400C00L	Total Lead	05/27/1992	07/30/1992	151		MG/KG	5.8	Moisture Content	07/10/1992	19.1	%WET W
SHA020240AB00L	Total Lead	05/27/1992	07/30/1992	106		MG/KG	5.6	Moisture Content	07/10/1992	15.2	%WET W
SHA0203100D00L	Total Lead	05/22/1992	07/29/1992	49.8	U	MG/KG	5.6	Moisture Content	07/09/1992	27.9	%WET W
SHA020310AC00L	Total Lead	05/22/1992	07/29/1992	92.9	U	MG/KG	5.1	Moisture Content	07/09/1992	20.9	%WET W

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TABLE 24
EAGLE PARK DATA SUMMARY

NI/TARACORP 89 ANALYTICAL REPORT

GENERATED: Sep 02, 1992

SAMPLE ID	PARAMETER	SAMPLE COLLECTION DATE	ANALYSIS DATE	RESULT	QUALIFIER	UNITS	REPORTING DETECTION LIMIT	PARAMETER	ANALYSIS DATE	RESULT	UNITS
SHA0203200D00L	Total Lead	05/22/1992	07/29/1992	101	J	MG/KG	6.1	Moisture Content	07/09/1992	23	%WET W
SHA020320AC00L	Total Lead	05/22/1992	07/29/1992	848	J	MG/KG	6.2	Moisture Content	07/09/1992	22.1	%WET W
SHA0203300D00L	Total Lead	05/22/1992	07/29/1992	1540	J	MG/KG	6.3	Moisture Content	07/09/1992	24.7	%WET W
SHA0203300D0LD	Total Lead	05/22/1992	07/29/1992	1220	J	MG/KG	6.3	Moisture Content	07/09/1992	23.7	%WET W
SHA0203300D00T	TCLP Lead	05/22/1992		0.54		MG/L	0.2				
SHA0203300E00L	Total Lead	05/22/1992	07/29/1992	507	J	MG/KG	6.9	Moisture Content	07/09/1992	29.8	%WET W
SHA0203300E00T	TCLP Lead	05/22/1992		0.31		MG/L	0.2				
SHA0203300F00L	Total Lead	05/22/1992	07/29/1992	95.9	J	MG/KG	6.6	Moisture Content	07/09/1992	30.8	%WET W
SHA0203400D00L	Total Lead	05/22/1992	07/29/1992	1800	J	MG/KG	7.1	Moisture Content	07/09/1992	34	%WET W
SHA0203400D00T	TCLP Lead	05/22/1992		< 0.20		MG/L	0.2				
SHA0203400E00L	Total Lead	05/22/1992	07/29/1992	148	J	MG/KG	7.1	Moisture Content	07/09/1992	30.8	%WET W
SHA0203400F00L	Total Lead	05/22/1992	07/29/1992	178	J	MG/KG	7.0	Moisture Content	07/09/1992	30.6	%WET W
SHA020340AC00L	Total Lead	05/22/1992	07/29/1992	186	J	MG/KG	5.9	Moisture Content	07/09/1992	21.8	%WET W
SHA0205100D00L	Total Lead	05/21/1992	07/29/1992	1030	J	MG/KG	6.5	Moisture Content	07/09/1992	24.8	%WET W
SHA0205100E00L	Total Lead	05/21/1992	07/29/1992	223	J	MG/KG	7.2	Moisture Content	07/09/1992	30.2	%WET W
SHA0205200D00L	Total Lead	05/21/1992	07/29/1992	529	J	MG/KG	5.9	Moisture Content	07/09/1992	18.4	%WET W
SHA0205200D0LD	Total Lead	05/21/1992	07/29/1992	832	J	MG/KG	6.1	Moisture Content	07/09/1992	21.3	%WET W
SHA0205200E00L	Total Lead	05/21/1992	07/29/1992	216	J	MG/KG	6.9	Moisture Content	07/09/1992	28.4	%WET W
SHA0205200F00L	Total Lead	05/21/1992	07/29/1992	20.4	J	MG/KG	6.4	Moisture Content	07/09/1992	25.9	%WET W
SHA0205300D00L	Total Lead	05/21/1992	07/29/1992	782	J	MG/KG	5.8	Moisture Content	07/09/1992	19.1	%WET W
SHA0205300D00T	TCLP Lead	05/21/1992		0.22		MG/L	0.2				
SHA0205300D0TD	TCLP Lead	05/21/1992		0.32		MG/L	0.2				
SHA0205300E00L	Total Lead	05/21/1992	07/29/1992	500	J	MG/KG	6.8	Moisture Content	07/09/1992	29.4	%WET W
SHA0205300E00T	TCLP Lead	05/21/1992		< 0.19		MG/L	0.19				
SHA020530AC00L	Total Lead	05/21/1992	07/29/1992	45	J	MG/KG	6.1	Moisture Content	07/09/1992	22.2	%WET W
SHI0100100C00L	Total Lead	05/20/1992	07/29/1992	1580	J	MG/KG	5.8	Moisture Content	07/08/1992	16.4	%WET W
SHI0100100D00L	Total Lead	05/20/1992	07/29/1992	843	J	MG/KG	6.1	Moisture Content	07/08/1992	18.8	%WET W
SHI010010AB00L	Total Lead	05/20/1992	07/29/1992	17900	J	MG/KG	51.1	Moisture Content	07/08/1992	7.9	%WET W
SHI010010AB00T	TCLP Lead	05/20/1992		152		MG/L	0.2				

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TABLE 24
EAGLE PARK DATA SUMMARY

NL/TARACORP 89MANALYTICAL REPORT

GENERATED: Sep 02, 1992

SAMPLE ID	PARAMETER	SAMPLE COLLECTION DATE	ANALYSIS DATE	RESULT	QUALIFIER	UNITS	REPORTING DETECTION LIMIT	PARAMETER	ANALYSIS DATE	RESULT	UNITS
SH#0100200C00L	Total Lead	05/20/1992	07/29/1992	90.2	J	MG/KG	5.2	Moisture Content	07/08/1992	20.5	%WET W
SH#010020AB00L	Total Lead	05/20/1992	07/29/1992	360	J	MG/KG	5.8	Moisture Content	07/08/1992	21.3	%WET W
SH#010020AB00T	TCLP Lead	05/20/1992		1.36		MG/L	0.2				
SRS0128100C00L	Total Lead	05/27/1992	07/30/1992	197		MG/KG	5.3	Moisture Content	07/10/1992	10	%WET W
SRS0128100D00L	Total Lead	05/27/1992	07/30/1992	1670		MG/KG	5.0	Moisture Content	07/10/1992	21.7	%WET W
SRS012810AB00L	Total Lead	05/27/1992	07/30/1992	53.2		MG/KG	5.2	Moisture Content	07/10/1992	6.4	%WET W
SRS0128200C00L	Total Lead	05/27/1992	07/30/1992	474		MG/KG	5.9	Moisture Content	07/10/1992	19.5	%WET W
SRS0128200D00L	Total Lead	05/27/1992	07/30/1992	163		MG/KG	5.4	Moisture Content	07/10/1992	12.6	%WET W
SRS0128200D00T	TCLP Lead	05/27/1992	09/15/1992	0.3		MG/L	0.18				
SRS0128200E00L	Total Lead	05/27/1992	07/30/1992	60.9		MG/KG	5.0	Moisture Content	07/10/1992	22.9	%WET W
SRS0128300C00L	Total Lead	05/27/1992	07/30/1992	745		MG/KG	5.5	Moisture Content	07/10/1992	13.3	%WET W
SRS0128300C00T	TCLP Lead	05/27/1992	09/15/1992	< 0.18		MG/L	0.18				
SRS0128300D00L	Total Lead	05/27/1992	07/30/1992	117		MG/KG	5.9	Moisture Content	07/10/1992	22.6	%WET W
SRS0128300D00T	TCLP Lead	05/27/1992		0.37							
SRS0128300E00L	Total Lead	05/27/1992	07/30/1992	57.2		MG/KG	5.4	Moisture Content	07/10/1992	22.1	%WET W
STE0203100C00L	Total Lead	05/20/1992	07/29/1992	10100	J	MG/KG	59.7	Moisture Content	07/08/1992	21.6	%WET W
STE0203100C0LD	Total Lead	05/20/1992	07/29/1992	5930	J	MG/KG	31.5	Moisture Content	07/08/1992	20.7	%WET W
STE0203100C00T	TCLP Lead	05/20/1992		71.6		MG/L					
STE0203100D00L	Total Lead	05/20/1992	07/29/1992	292	J	MG/KG	5.3	Moisture Content	07/08/1992	24.6	%WET W
STE020310AB00L	Total Lead	05/20/1992	07/29/1992	45200	J	MG/KG	105	Moisture Content	07/08/1992	6.1	%WET W
STE020310AB0LD	Total Lead	05/20/1992	07/29/1992	37700	J	MG/KG	108	Moisture Content	07/08/1992	7.4	%WET W
STE020310AB00T	TCLP Lead	05/20/1992		156		MG/L					
STE0203200C00L	Total Lead	05/20/1992	07/29/1992	820	J	MG/KG	5.7	Moisture Content	07/08/1992	16.7	%WET W
STE0203200D00L	Total Lead	05/20/1992	07/29/1992	44.2	J	MG/KG	5.1	Moisture Content	07/08/1992	21.5	%WET W
STE020320AB00L	Total Lead	05/20/1992	07/29/1992	8060	J	MG/KG	26.1	Moisture Content	07/08/1992	5.3	%WET W
STE020320AB00T	TCLP Lead	05/20/1992		52.3		MG/L	0.2				
STE0203300C00L	Total Lead	05/20/1992	07/29/1992	126	J	MG/KG	5.1	Moisture Content	07/08/1992	21	%WET W
STE0203300D00L	Total Lead	05/20/1992	07/29/1992	41.5	J	MG/KG	5.1	Moisture Content	07/08/1992	23.5	%WET W
STE020330AB00L	Total Lead	05/20/1992	07/29/1992	5430	J	MG/KG	28.1	Moisture Content	07/08/1992	12.9	%WET W
STE020330AB0LD	Total Lead	05/20/1992	07/29/1992	9140	J	MG/KG	28.5	Moisture Content	07/08/1992	13.3	%WET W

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TABLE 24
EAGLE PARK DATA SUMMARY

NI/TARACORP 89MANALYTICAL REPORT

GENERATED: Sep 02, 1992

SAMPLE ID	PARAMETER	SAMPLE COLLECTION DATE	ANALYSIS DATE	RESULT	QUALIFIER	UNITS	REPORTING DETECTION LIMIT	PARAMETER	ANALYSIS DATE	RESULT	UNITS
STE020330AB05/20	TCLP Lead	05/20/1992		32.2		MG/L	0.2				
STE0203400C00L	Total Lead	05/20/1992	07/29/1992	971 U		MG/KG	5.5	Moisture Content	07/08/1992	23.4	%WET W
STE0203400D00L	Total Lead	05/20/1992	07/29/1992	59.8 U		MG/KG	5.6	Moisture Content	07/08/1992	25.2	%WET W
STE020340AB00L	Total Lead	05/20/1992	07/29/1992	37500 U		MG/KG	101	Moisture Content	07/08/1992	7.5	%WET W
STE020340AB00T	TCLP Lead	05/20/1992		101		MG/L	0.2				
STE0208100C00L	Total Lead	05/21/1992	07/29/1992	52 U		MG/KG	5.1	Moisture Content	07/09/1992	21.3	%WET W
STE020810AB00L	Total Lead	05/21/1992	07/29/1992	2170 U		MG/KG	5.1	Moisture Content	07/08/1992	21.9	%WET W
STE020810AB00T	TCLP Lead	05/21/1992		1.79		MG/L	0.2				
STE0208200C00L	Total Lead	05/21/1992	07/29/1992	88.9 U		MG/KG	5.3	Moisture Content	07/09/1992	21.7	%WET W
STE020820AB00L	Total Lead	05/21/1992	07/29/1992	474 U		MG/KG	5.2	Moisture Content	07/09/1992	23.5	%WET W
STE020820AB00T	TCLP Lead	05/21/1992		0.88		MG/L	0.2				
STE0208300C00L	Total Lead	05/21/1992	07/29/1992	19.4 U		MG/KG	5.7	Moisture Content	07/09/1992	19.4	%WET W
STE020830AB00L	Total Lead	05/21/1992	07/29/1992	90.7 U		MG/KG	5.8	Moisture Content	07/09/1992	26.4	%WET W
STE0208400C00L	Total Lead	05/21/1992	07/29/1992	2100 U		MG/KG	5.1	Moisture Content	07/09/1992	20.6	%WET W
STE020840AB00L	Total Lead	05/21/1992	07/29/1992	2790 U		MG/KG	29.7	Moisture Content	07/09/1992	22.9	%WET W
STE020840AB00T	TCLP Lead	05/21/1992		0.51		MG/L	0.2				
STE0208500C00L	Total Lead	05/21/1992	07/29/1992	4070 U		MG/KG	27.3	Moisture Content	07/09/1992	16.9	%WET W
STE020850AB00L	Total Lead	05/21/1992	07/29/1992	1180 U		MG/KG	5.3	Moisture Content	07/09/1992	24.3	%WET W
STE020850AB00T	TCLP Lead	05/21/1992		0.53		MG/L	0.2				

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TABLE 28
2230 CLEVELAND DATA SUMMARY

NL/TARACORP 89MC114V

ANALYTICAL REPORT

SAMPLE ID	PARAMETER	SAMPLE COLLECTION DATE	ANALYSIS DATE	RESULT	QUALIFIER	UNITS	REPORTING DETECTION LIMIT	PARAMETER	ANALYSIS DATE	RESULT	UNITS
SCL2230100A00L	Total Lead	04/22/1992	06/03/1992	525		MG/KG -	5.8	Moisture Content	06/10/1992	19.4	%WET W
SCL2230100B00L	Total Lead	04/22/1992	06/03/1992	422		MG/KG -	6.2	Moisture Content	06/10/1992	19.4	%WET W
SCL2230100C00L	Total Lead	04/22/1992	06/03/1992	148		MG/KG -	5.9	Moisture Content	06/10/1992	18.6	%WET W
SCL2230200A00L	Total Lead	04/22/1992	06/03/1992	1020		MG/KG -	6.2	Moisture Content	06/10/1992	20.9	%WET W
SCL2230200B00L	Total Lead	04/22/1992	06/03/1992	613		MG/KG -	6.1	Moisture Content	06/10/1992	19.1	%WET W
SCL2230200C00L	Total Lead	04/22/1992	06/03/1992	433		MG/KG -	6.0	Moisture Content	06/10/1992	18.7	%WET W
SOR0001100A00T	TCLP Lead	04/22/1992		10.3		MG/L					
SOR0001100A0TD	TCLP Lead	04/22/1992		11.2		MG/L					
SOR0002100A00T	TCLP Lead	04/22/1992		72.8		MG/L					
SOR0003100A00T	TCLP Lead	04/22/1992		15.6		MG/L					

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TABLE 29
3108 COLGATE DATA SUMMARY

NL/TARACORP 89MCANALYTICAL REPORT GENERATED: Sep 09, 1992

SAMPLE ID	PARAMETER	SAMPLE COLLECTION DATE	ANALYSIS DATE	RESULT (ppm)	QUALIFIER	UNITS	REPORTING DETECTION LIMIT	PARAMETER	ANALYSIS DATE	RESULT	UNITS
SOR0026100A00L	Total Lead	05/13/1992	07/28/1992	3390	J	MG/KG -	26.5	Moisture Content	07/05/1992	9.3	%WET W
SOR0026100B00L	Total Lead	05/13/1992	06/26/1992	11900		MG/KG -	53.4	Moisture Content	07/05/1992	13.3	%WET W
SOR0026100C00T	TCLP Lead	05/13/1992		10.9		MG/L					
SOR0026200A00L	Total Lead	05/13/1992	06/26/1992	81.1		MG/KG -	5.2	Moisture Content	07/05/1992	9.6	%WET W
SOR0026200B00L	Total Lead	05/13/1992	06/26/1992	70.1		MG/KG -	5.5	Moisture Content	07/05/1992	11.5	%WET W
SOR0026200C00L	Total Lead	05/13/1992	06/26/1992	64.9		MG/KG -	5.5	Moisture Content	07/05/1992	12.9	%WET W

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TABLE 30
1628 DELMAR AVENUE DATA SUMMARY

NL/TARACORP 89MC114V

ANALYTICAL REPORT GENERATED: Sep 10, 1992

SAMPLE ID	PARAMETER	SAMPLE COLLECTION DATE	ANALYSIS DATE	RESULT	QUALIFIER	UNITS	REPORTING DETECTION LIMIT	PARAMETER	ANALYSIS DATE	RESULT	UNITS
SDE1628100A00L	Total Lead	03/03/1992	03/18/1992	1620		MG/KG -	5.9	Moisture Content	03/05/1992	18.4	%WET W
SDE1628100A0LD	Total Lead	03/03/1992	03/18/1992	1730		MG/KG -	6.0	Moisture Content	03/05/1992	18	%WET W
SDE1628100B00L	Total Lead	03/03/1992	03/18/1992	722		MG/KG -	5.7	Moisture Content	03/05/1992	17	%WET W
SDE1628100B0LD	Total Lead	03/03/1992	03/18/1992	680		MG/KG -	5.8	Moisture Content	03/05/1992	17.5	%WET W
SDE1628100C00L	Total Lead	03/03/1992	03/18/1992	278		MG/KG -	5.7	Moisture Content	03/05/1992	16.5	%WET W
SDE1628100C0LD	Total Lead	03/03/1992	03/18/1992	280		MG/KG -	5.5	Moisture Content	03/05/1992	16.7	%WET W
SDE1628200A00L	Total Lead	03/03/1992	03/18/1992	1250		MG/KG -	6.2	Moisture Content	03/05/1992	22.7	%WET W
SDE1628200B00L	Total Lead	03/03/1992	03/18/1992	833		MG/KG -	6.3	Moisture Content	03/05/1992	22.4	%WET W
SDE1628200C00L	Total Lead	03/03/1992	03/18/1992	107		MG/KG -	6.4	Moisture Content	03/05/1992	22.5	%WET W
SOR0025300A00T	TCLP Lead	05/13/1992		0.47		MGL					
SOR0025400A00T	TCLP Lead	05/13/1992		0.11		MGL					

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TABLE 25
MISSOURI AVENUE DATA SUMMARY

NL/TARACORP 89MC114V

ANALYTICAL REPORT GENERATED: Sep 09, 1992

SAMPLE ID	PARAMETER	SAMPLE COLLECTION DATE	ANALYSIS DATE	RESULT (ppm)	QUALIFIER	UNITS	REPORTING DETECTION LIMIT
SOR000710AB00T	TCLP Lead	12/10/1991	01/07/1992	180		MG/L	0.65
SOR000810AB00T	TCLP Lead	12/10/1991	01/07/1992	<0.65		MG/L	0.65
SOR000910AB00T	TCLP Lead	12/10/1991	01/07/1992	235		MG/L	0.65
SOR001010AB00T	TCLP Lead	12/10/1991	01/07/1992	82.5		MG/L	0.65
SOR0013100K00T	TCLP Lead	06/29/1992		3.94		MG/L	
SOR0014100K00T	TCLP Lead	06/29/1992		< 0.17		MG/L	
SOR0015100J00T	TCLP Lead	06/29/1992		< 0.19		MG/L	
SOR0015100K00T	TCLP Lead	06/29/1992		0.68		MG/L	

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EXHIBIT II

OPERATING MANUAL FOR THE SPECTRACE 9000
PORTABLE XRF ANALYZER

1

Spectrace 9000

Portable XRF Analyzer

Part No. 717715
Rev. 0.3
Preliminary
January 1992

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Contents

Chapter 1: Introduction	1-1
Components	1-1
Analytical Capabilities	1-2
Software	1-2
Operational Modes	1-2
Chapter 2: Basic Operation	2-1
Assembly	2-1
Start Up	2-2
Chapter 3: Sample Presentation and Preparation	3-1
Sample Cups	3-2
In-situ Analysis	3-3
Sample Preparation	3-3
Solids	3-3
Soils and Other Heterogenous Materials	3-4
Chapter 4: Software	4-1
The Keyboard	4-1
Start-Up	4-1
The Main Menu	4-2
1 Measure (Ready Screen)	4-2
2 Choose an application	4-3
3 Review stored results	4-3
4 Review stored spectra	4-4
5 More (Other Functions Menu)	4-5
Ready Screen (Measurement)	4-6
Measure	4-6
Modify	4-8
Options	4-9
Spectrum Display	4-11
Flow Charts	4-13
Main Menu	4-13
Ready Screen	4-14
Other Functions	4-15
Examine a Spectrum	4-16
Ready Screen Options	4-17

Chapter 5: Maintenance	5-1
Main Power Battery	5-1
Probe Detector Bias Battery	5-1
Probe Window Replacement.....	5-3
 Chapter 6: Radiation Safety	 6-1
Routine Precautions	6-1
In Case of Malfunction	6-2
Software Failure	6-2
Hardware Failure	6-2
Problem Prevention.....	6-3
Safety Provisions in the Instrument Design	6-3
The Radiation Sources	6-4
Source Capsule Design	6-4
Radiation Levels	6-5
 Chapter 7: Regulatory Requirements.....	 7-1
Licensing	7-1
Leak Testing	7-1
Transportation	7-1
Additional Safety Items	7-2
 Glossary	 G-1
 Appendix: General Licenses	 A-1
Environmental and Technical Services	A-17
 Index	 i

Specifications

Main Components

Probe	High-resolution mercuric iodide detector and radioisotope excitation sources
Electronics Unit	Data acquisition, processing, and display unit
Cable	Interconnecting cable (2 meter length)
Application Generator	IBM™ compatible PC/AT™ software package
Carrying/Shipping case	Heavy duty industrial grade with foam inserts

Analytical Capabilities

Element Range	77 elements, sulfur through uranium (Z=16 through 92)
Minimum Detection Limit (MDL)	50-100 ppm for most of the elements in the range of analysis
Measurement Time	Typically 30-200 seconds
Number of Elements	Up to 25 in each Application
Number of Applications	Up to 50 Applications can be stored in the analyzer and selected by user-defined name as needed

Electronics Unit

Operating Temperature	Standard: 0°C to 55°C (32°F to 130°F) Extended (optional): -10°C to 70°C (14°F to 158°F)
Storage Temperature	Standard: -20°C to 60°C (-4°F to 140°F) Extended (optional): -40°C to 80°C (-40°F to 175°F)
Dimensions	32 cm x 30 cm x 10 cm (13 in x 12 in x 4 in)
Weight	6.7 kg (14.75 lbs) with batteries
Connectors	25-pin D connector for RS-232C serial I/O Receptacle for AC adaptor/battery charger Probe cable connector

Electrical	Operation from battery or AC line (110/220 VAC, 50-60 Hz)
Display	High contrast, wide-angle view LCD 240x64 graphics resolution 30 column x 8 row alphanumeric characters Size: 13 cm width x 3.6 cm height (5.1 in x 1.4 in) Adjustable screen contrast
Controls	Sealed 21-key keypad

Probe**3 x-ray excitation sources**

²⁴¹Am	Measures elements Cu to Tm (K x-rays); W to U (L x-rays)
¹⁰⁹Cd	Measures elements Ca to Rh (K x-rays); Ba to U (L x-rays)
⁵⁷Fe	Ta to Pb (K x-rays) Measures elements S to Cr (K x-rays); Mo to Ba (L x-rays)

The analyzer software automatically selects which sources to use and the measurement time for each source based on the stored information for each application

X-ray detector	High resolution HgI ₂ (mercuric iodide)
Operating Temperature	-10°C to 49°C (14°F to 120°F)
Storage Temperature	-40°C to 43°C (-40°F to 110°F)
Housing	Splash proof, soap and water washable
Dimensions	12.7 cm x 7.6 cm x 21.6 cm (5 in x 3 in x 8.5 in)
Weight	1.9 kg (4.2 lbs)
Controls	Start button

Index

"<—" key 4-1

A

accessories 1-1
acquire background data 4-9
adapter ring (30mm) 3-2
adjust calibration 4-10
agreement state 7-1
Am-241 1-2
Application
 library 1-2, 4-3
 maintenance 4-5
 selection 4-3
auto scaling 4-11
autocycle 4-7, 4-10
auxiliary batteries 5-1

B

background data 4-9
Batteries
 auxiliary 5-1
 battery charger 2-1
 discharged battery 5-1
 disconnected (when shipped) 2-1
 lithium cell 5-2
 low battery 5-1
 main power 5-1
 charging 5-1
 probe detector bias 5-1
 sensing circuit 5-1
 replacement 5-1
bias supply 5-1

C

Calibration
 adjust 4-10
 restore original 4-10
Cd-109 1-2
CHAN/DOT 4-11
channel number 4-12
choose application 4-3
COM port setup
 default 4-5
computer link 4-5
"CONT/PAUSE" 4-1
contrast knob 2-2
count time 4-2, 4-8
cyclic operation 4-7, 4-10

D

Default
 calibration 4-10
 COM port setup 4-5
Delete
 stored results 4-3
 stored spectra 4-4
detection limit 1-2
detector bias battery 5-1
disable/enable element lines 4-12
discharged battery 5-1
display contrast 2-2
display thresholds 4-8, 4-10
disposal 7-2, A-17

E

edit key 4-1
electronics unit 1-1
element lines 4-12
element range 1-2
emergencies. *See Radiation Safety*
end autocycling 4-7
energy calibration 4-9, 4-12
environmental and technical services division A-17
examine spectrum 4-5, 4-7, 4-10

F

fail safe provisions 6-4
Fe-55 1-2
FINE/COARS 4-12
first screen 4-1
flashing light red/yellow 4-6, 5-1
flow charts 4-13
fundamental parameter XRF 1-2

G

general license 7-1, A-4, A-4-A-5

H

half-life 6-4
hardware failure 6-2
heterogenous materials 3-4
Hmag (horizontal magnification) 4-11

I

in-situ analysis 3-1, 3-3
initialization routines 4-1
inspections A-9
instrument design 6-3

K

keyboard 4-1

L

lab stand 3-1
 last results (show) 4-9
 leak test records A-5
 leak tests 7-1, A-2, A-4, A-10, A-17
 Licensing
 leak test records A-5
 leak tests A-4
 license obligations A-4
 sale or transfer of unit A-5
 theft or loss of unit A-11
 licensing 7-1, A-13-A-16
 licensing agencies A-13
 licensing and regulatory affairs A-17
 light flashing red/yellow 4-6, 5-1
 lithium cell 5-2
 live/real time 4-8
 log display 4-11
 lost radioactive material A-3
 low battery condition 5-1

M

Main Menu 2-2, 4-1, 4-2
 flow chart 4-13
 Other Functions flow chart 4-15
 malfunctions 6-2
 Measure (Ready Screen) 4-2, 4-6
 measuring 4-6
 mercuric iodide detector 1-1
 Modify screen 4-8
 moisture content (of soil sample) 3-3

N

negative offset 4-10
 negative results 4-8, 4-10
 NRC 7-1

O

O-ring (in probe) 5-4
 offset, negative 4-10

P

packing list 2-1
 particle size 3-3
 parts (included) 2-1

PgUP, PgDN 4-3
 power, main battery 5-1
 precautions, radiation safety 6-1
 preparation, samples 3-3
 printer link 4-5
 probe 1-1
 probe button 4-6
 probe detector bias battery 5-1
 probe window replacement 5-3
 problems
 hardware failure 6-2
 software failure 6-2
 "source did not close" message 6-2

R

radiation
 field 6-3
 records A-2
 Radiation Safety 4-6, 6-1
 accidents 7-2
 disposal 7-2
 exposure risk 6-1
 fail safe 6-4
 instrument design 6-3
 malfunction 6-2
 radiation levels 6-5
 radiation sources 6-4
 radiation warning 4-6
 routine precautions 6-1
 shipping radioactive material A-17
 source capsule design 6-4
 training A-17
 transporting unit 7-1
 radioactive material A-6
 emergency shipments A-7
 theft or loss A-11
 transfer of A-6
 radioisotope sources 1-2
 RAM memory 4-1
 ranges of elements 1-2
 raw data 4-7
 Ready Screen 4-2
 flow chart 4-14
 options flow chart 4-17
 real/live time 4-8
 Recall
 spectra and recal 4-9
 stored results 4-3
 stored spectra 4-4

regulatory requirements 7-1
repetitive measurement 4-7
restore original calibration 4-10

Results

delete 4-3
storage capacity 1-1
store/send automatically 4-5
stored 4-3
thresholds 4-8, 4-10
to send 4-6
to store 4-7

Results Screen 4-6

options 4-7

S

sale or transfer of unit A-5

sample cups 3-2

sample ID 4-6

Samples

heterogenous materials 3-4
ID (labelling for storage) 4-6
large solids 3-1
moisture content 3-3
particle size 3-3
preparation 3-3
presentation 3-1
small amount 3-3
soil 3-3
surface preparation 3-3
unprepared 3-4

sampling errors 3-4

scroll 4-3

send/store mode 4-5

sensing circuit 5-1

set clock/calendar 4-5

sets of spectra 4-4

shield 6-1, 6-3

shield cup 3-1

shipping radioactive material A-2

small sample 3-3

Software 4-1

version number 2-2, 4-1

software failure 6-2

soil samples 3-3

Source 6-4

accidental exposure 6-3

activity and half life 6-3

capsule design 6-4

capsules 6-3

"did not close" message 6-2

holders 6-4

source decay 2-2

SOURCE ON indicators 6-1

when exposed 6-3

source-decay compensation 4-1

sources 1-2

specific license 7-1

Spectra

flow chart 4-16

review 4-4

storage capacity 1-1

store/send automatically 4-5

stored 4-4

to examine 4-7

to send 4-7

to store 4-7

Spectrum Display 4-11

standard deviation/units 4-7

start-up 4-1

store/send mode 4-5

T

theft or loss A-11

time and date 2-2, 4-1

Total Measurement Time 4-8

transmit reports or spectra 4-6

transporting unit 7-1

U

units/standard deviation 4-7

unpack (upon delivery) 2-1

unprepared samples 3-4

V

version number 4-1

Vmag (vertical magnification) 4-11

W

window plate 5-3

window, probe 5-3

window replacement 5-3

Z

Zeroing

current result 4-8

examine and modify zeros 4-9

on a blank 4-9

Chapter 1: Introduction

X-radiation of sufficient energy will cause all atoms to *fluoresce*, emitting x-rays of characteristic energy (wavelength). By analyzing the fluorescent radiation emitted by a sample under excitation, both the identity and the quantity of the elements present in the sample can be determined.

The Spectrace 9000 is an x-ray fluorescence analyzer containing radioisotope sources to provide the necessary excitation and a high-resolution x-ray detector for the resulting fluorescence. It consists of two main components: a probe and an electronics unit.

Caution: *The probe contains radioactive material. Before using this instrument it is essential that you review the radiation safety procedures and information in Chapter 6: Radiation Safety.*

Components

Probe

The probe consists of a sealed aluminum enclosure containing a high resolution mercuric iodide detector and three radioisotope x-ray excitation sources. The probe aperture, through which the analysis is performed, is sealed with a thin, replaceable plastic film. The probe also contains a pre-amplifier and bias supply for the detector and a mechanism to move the radioisotope sources from their shielded location during an analysis.

Electronics Unit

The electronics unit provides data acquisition, processing, and display capabilities. Its computer includes a math coprocessor for fast calculation of results. Sufficient memory is available to store up to 300 sets of analysis results and up to 120 spectra. An RS-232 port allows stored data to be transferred to another computer. The graphics display allows direct viewing and qualitative analysis of the x-ray spectra. The replaceable and rechargeable internal battery provides for field-portable operation.

Accessories

The system is supplied with an industrial grade carrying case, a lab stand for bench-top use of the probe, and a sample cup kit for presentation of powdered and liquid samples.

Analytical Capabilities

The Spectrace 9000 uses three radioisotope sources. Each source emits a different energy (wavelength) of radiation which provides efficient analysis of specific ranges of elements:

<i>Source</i>	<i>Element Range</i>
Fe-55	S to Cr (K x-rays) and Mo to Ba (L x-rays)
Cd-109	Ca to Rh (K x-rays) Ta to Pb (K x-rays) and Ba to U (L x-rays)
Am-241	Cu to Tm (K x-rays) and W to U (L x-rays)

With these three sources, the Spectrace 9000 can effectively analyze any element above sulfur in the periodic table. The detection limit is typically 50-100 ppm for most analytes.

Software

The Spectrace 9000 utilizes a fundamental parameter XRF calibration derived from theoretical considerations (as opposed to empirical data). The menu driven software in the Spectrace 9000 supports multiple XRF calibrations called "Applications." Each Application is a complete analysis configuration including elements to be measured, interfering elements in the sample, and a set of fundamental parameter calibration coefficients.

Operational Modes

In-situ analysis of a wide variety of solid materials is possible by placing the Spectrace 9000 probe directly in contact with the sample.

Other materials such as liquids, powders, and pastes can be analyzed with little or no preparation using the sample cups. See *Chapter 3: Sample Presentation and Preparation* for a complete discussion.

Chapter 2: Basic Operation

Unpack everything from the case. Check to make sure that you have all the necessary parts:

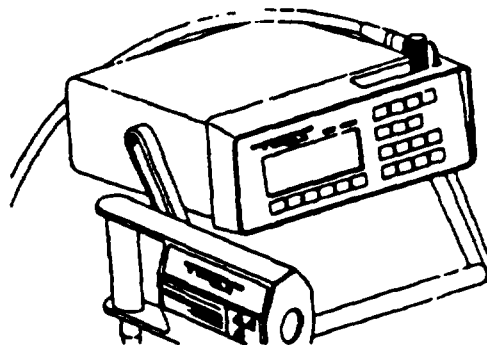
- Electronics unit
- Hand held probe with safety cover and one (1) spare window assembly
- Interconnecting cable (2 meters)
- Two (2) blank check samples
- Probe laboratory stand for table top use, safety shield, and sample adapter ring for standard 30 mm x-ray sample cups
- Battery charger
- Instruction manual
- RS-232C interface cable (2.3 meters)
- Two (2) screwdrivers for changing battery and probe window
- Shoulder strap
- Carrying/shipping case
- Spare probe battery (AA size)

Assembly

Note: The unit is shipped with the battery disconnected.

To connect the battery, set the electronics unit on its face and use the flat blade screwdriver provided to loosen the two 1/4-turn fasteners on the back. Remove the battery pack. Inside, find the cord with the red cap (it covers a three-pronged plug). Remove the cap and plug it into the battery pack. Put the pack back into the unit—it can only fit one way. Tighten the fasteners.

Set the electronics up with the handle latched in the bench-top position. Carefully plug the right angle end of the probe cable into the electronics unit and the other end into the probe handle.



If desired, place the probe in the lab stand at this time. If the unit is to be used on the bench for an extended period, use the battery charger provided. (It plugs in right next to the probe cable on the electronics unit.)

Start Up

Press the ON key. In two or three seconds, the display announces the version number of the software. If necessary, adjust the contrast knob located on the underside of the front bezel. This knob can be turned so far that the display appears blank.

The initial screen will display for about ten seconds and then the unit will ask if the time and date are set correctly.

Note: The date must be set correctly otherwise large errors in source-decay compensation can result. Furthermore, reports include time and date of analysis; incorrect settings may cause administrative confusion.

After the time and date screens, the Main Menu will appear:

MAIN MENU					
1	Measure				
2	Choose an application				
3	Review stored results				
4	Review stored spectra				
5	More_				
1	2	3	4	5	6

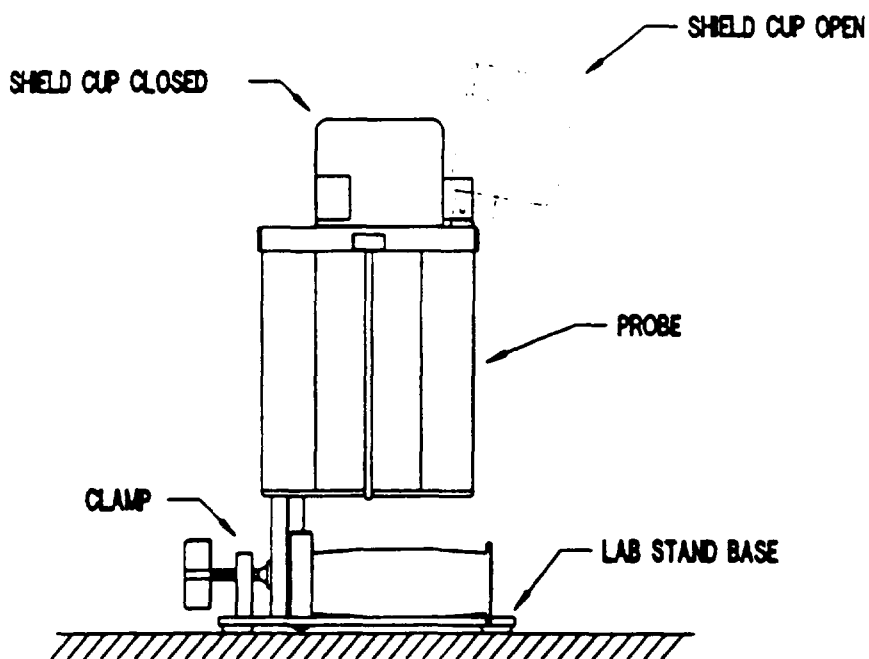
The Main Menu offers several selections. Each of these is discussed in detail in *Chapter 4: Software*. Before attempting any serious analytical work, be sure to read *Chapter 3: Sample Presentation and Preparation*.

Chapter 3: Sample Presentation and Preparation

Sample *presentation* is the positioning of the sample with respect to the probe aperture. Proper and consistent presentation is essential for accurate analytical results.

Ideally, the sample should be flat, larger than 1" in diameter, and should be placed in contact with the face of the probe. For flat metal plates, sheets of plastic and paper this is easily achieved. Other sample forms such as liquids or powders must be contained for presentation to the probe.

Large solid samples are most easily analyzed by placing the probe directly on the sample (in-situ analysis). For small samples it is usually more convenient to set up the unit on a table with the probe in the lab stand. The lab stand holds the probe with the aperture facing directly upwards so samples can be placed in position. Samples must cover the probe window. A shield cup that will stop any radiation transmitted through thin samples is provided for use with this configuration.



Sample Cups

Liquids and powders should be presented using sample cups. These cups are made up of three pieces: a cup, a ring and a piece of Mylar film.

To load a sample cup:

1. Fill the cup nearly full with your sample. If the sample is powdered, tap the cup a little to settle the contents and, if necessary, add more sample until the cup is at least 3/4 full.
2. Tear off a square (more or less) piece of the film. Lay it over the open cup approximately centered.
3. Examine the sample cup ring. One inside edge is rather sharp and the other inside edge is definitely smooth and rounded. Place the ring over the cup with the rounded edge down.
4. Using your thumbs and forefingers (or three fingers of one hand), press the ring down slowly and evenly until it is flush with the end of the cup. If properly done, the ring will slide down and snap into position leaving the film taut and wrinkle-free. If you do get wrinkles, sometimes they can be removed by pulling on the excess film in the appropriate places. Occasionally the window comes out so wrinkled you just have to take the ring off and start over with a new piece of film.

Note: Wrinkles cause part of the sample to be held away from the face of the probe and can interfere with analysis. For maximum accuracy the film must be taut and free of wrinkles.

5. Turn the filled and sealed sample cup upside down and, if the sample is powdered, tap the cup on the bench to thoroughly settle and compact the contents.

The sample is now ready to be placed film side down on the probe for analysis. The 30mm adapter ring should be placed in the large hole in the shield cup base. This adapter ring locates the sample cup reproducibly over the probe aperture.

Note: Make certain the sample cup rests in contact with the probe face, otherwise significant analysis errors may result.

In-situ Analysis

In-situ analysis is appropriate for soils, manufactured items, and large objects. In other words, anytime it's easier to take the analyzer to the sample than it is to bring the sample to the analyzer.

Note: The probe face must be kept in contact with the sample throughout the analysis. Whenever possible, arrange the sample so the probe can be set down on top of it.

In the case of in-situ soil analysis, best results are obtained on reasonably dry, flat, compacted surfaces of fine-grained soils. Whenever possible, flatten and compact the area to be measured with your foot or an appropriate tool. Good results can be obtained at moisture contents up to about 25% (beyond this point the soil is wet mud and must be contained in a sample cup). In-situ analysis of wet mud will grossly contaminate the probe window, invalidating all subsequent measurements until the window is cleaned.

Caution: *The aperture window can be punctured. Clear test areas of sharp, hard or protruding objects (for example, twigs or rocks). Failure to clear the test area can result in damage to the instrument.*

Coarse-grained soil conditions may not permit a truly representative sample and may adversely affect the analysis results. Such samples should be prepared before analysis.

Sample Preparation

Sample preparation is the treatment given to the "as received" sample to make it suitable for XRF analysis. Most samples require little or no sample preparation. Homogeneous solids, clear solutions and finely ground powders (<200 mesh) can be accurately analyzed with no preparation (other than filling the sample cup).

Solids

Solid samples (sheets of metal, plastic or paper, for examples) can usually be analyzed without preparation. The size of the sample will determine whether you use the lab stand or in-situ analysis. The analyzed surface of the sample should be relatively clean, since many element's x-rays will not penetrate a thick layer of dirt. (Of course, if the sample is a thick layer of dirt it should remain, well, dirty.)

Note: The sample should cover the aperture completely. If you have a very small sample which does not cover the aperture completely, please call us for assistance.

Soils and Other Heterogenous Materials

The highly variable nature of soils increases the possibility of XRF analysis errors. It is difficult to predict whether these errors will make the results too high or too low. We recommend that whenever possible a comparison be made between the XRF results on prepared and unprepared material. For field screening work, this can be accomplished by collecting samples from a representative number of the locations analyzed. These samples should be dried and ground, and presented in sample cups. If desired, they can also be split *after* drying and grinding for chemical analysis.

Note: Do not split a small sample of coarse material. The splits will not be representative.

This comparison will show either:

- the unprepared sample results agree satisfactorily with both the chemistry and the XRF results on the prepared samples; or
- an appropriate correction factor should be applied to unprepared sample results; or
- the errors introduced by the variables in the unprepared material are too large to tolerate. Sample preparation will be required.

Chapter 4: Software

This chapter is a reference guide to the screens and options presented by the analyzer software. Each screen, and each option on each screen, is discussed in logical order. At the end of this chapter are flow charts which outline the menu structure of the Spectrace 9000 software.

The menus for routine operations are designed to be easy to use and understand. However, it is easy to become confused when dealing with such a powerful system. Please call us for assistance with any problem you may encounter.

The Keyboard

The row of keys under the LCD screen perform functions defined by labels that the software writes to the bottom line of the display. As you move through the various menus, these keys are redefined to provide an efficient user interface.

The keypad to the right of the screen is used for numeric entry.

The "CONT/PAUSE" key (referred to as CONT) is used

- as an ENTER key
- to begin an analysis
- to pause an analysis in progress.

The "<—" key is used to edit entries before pressing CONT.

Start-Up

The first screen to appear when you turn the instrument on shows the version number of the software. This screen displays for about 10 seconds while a number of initialization routines are executed. Next the RAM memory size is announced (usually 512K) and then the time and date screens appear. Time and date screens are in the flow chart on page 4-13.

Caution: *The first time you turn on the unit after connecting the battery, the time and date MUST be set. It is critical that the date be accurate, since source-decay compensation is based on it.*

The Main Menu is the next screen to appear after the time and date screens.

The Main Menu

MAIN MENU					
1 Measure					
2 Choose an application					
3 Review stored results					
4 Review stored spectra					
5 More_					
1	2	3	4	5	6

A flow chart of the Main Menu is on page 4-13.

1 Measure (Ready Screen)

This Main Menu selection takes you to the screen shown below. This screen is referred to as the Ready Screen.

SOIL ANALYSIS					
updated 18NOV91					

Analysis Time L			TO MEASURE:		
Cd	Fe	Am	Press CONT or		
200	200	200	probe button.		
-----			-----		
Modify_			Options Quit		
1	2	3	4	5	6

The Ready Screen shows the Application name (with some description), the count times for each of the three sources, and accesses other options. (See *Ready Screen (Measurement)* on page 4-6 for detailed discussion.)

A flow chart of the Ready Screen begins on page 4-14.

2 Choose an application

This Main Menu selection lists the Applications currently loaded in the unit.

Application library index.					
> SOIL ANALYSIS					
THIN SAMPLE ANALYSIS					
UP	DOWN	OK	EXPND	QUIT	
1	2	3	4	5	6

UP and DOWN scroll the Application list past the ">" pointer (on the left side of the screen).

OK selects the Application at the ">" pointer.

EXPND shows the entire description of the Application.

QUIT returns you to the Main Menu.

3 Review stored results

This Main Menu selection lists the stored results.

Note: UP and DOWN scroll is used on many screens. Where UP and DOWN are displayed, pressing the "0" key will toggle to PgUP and PgDN for rapid movement through long lists (press 0 again to return to UP and DOWN).

Results are stored for:					
>25NOV91 11:36:26 TEST					
UP	DOWN	DEL	VIEW ALL	QUIT	
1	2	3	4	5	6

DEL deletes the ">" (selected) result from memory. A confirmation message, "Are you sure?" will display.

VIEW shows you the Results Screen for that set of results.

ALL brings up a new screen, which enables you to either send all stored results out the COM port or delete all stored results.

QUIT returns you to the Main Menu.

4 Review stored spectra

This Main Menu selection functions similarly to the previous one, except it operates on stored sets of spectra. Each measurement usually has a set of three spectra.

Spectra are stored for:

>21NOV91 14:37:25 B

UP	DOWN	DEL	SEND	ALL	QUIT
<u>1</u>	2	3	4	5	6

DEL deletes the "> " (selected) result from memory.

SEND sends the selected set of spectra out the COM port.

ALL brings up a new screen, which enables you to either send all stored spectra out the COM port or delete all stored spectra.

QUIT returns you to the Main Menu.

5 More (Other Functions Menu)

The last Main Menu lists other functions.

- 1 **Set Clock/Calendar** Lets you examine or reset the system time and date.
- 2 **Comm. port setup** Shows you the current COM port configuration and allows MODification of baud rate, word length, parity, and XON-XOFF status. There are two configurations: the "printer link" is for sending stored results and spectra to either a printer or another computer. The "computer link" is exclusively for loading new Applications using the special Application Generator PC software.

The default COM setup for both is 9600,8,N,1.
- 3 **Set store/send modes** Allows you to specify automatic sending for any combination of results, intensities, and spectra to a printer or computer. The store mode enables automatic on-board storing of results and/or spectra.
- 4 **Application maintenance** Lists functions pertaining to selecting, deleting, and examining Applications. Also provides a way to transfer new Applications from a PC (an operation that is guided and supervised by the PC software).
- 5 **Examine spectrum** Allows you to select a single stored spectrum or collect a new spectrum and display it. See *Spectrum Display*, page 4-11, for more information.

A flow chart of the Other Functions Menu is on page 4-15.

Ready Screen (Measurement)

(This details the *1 Measure (Ready Screen)* of the Main Menu, as referenced on page 4-2.) A flow chart of the Ready Screen is on page 4-14.

There are four actions you can select from the Ready Screen:

- Measure
- Modify
- Options
- Quit

Measure

Warning: During analysis, there is a minimal risk of minor radiation exposure. While an analysis is in progress:
Do not remove a sample from the probe aperture,
Do not look into the aperture,
Do not touch the aperture.

Press either CONT or the probe button to start an analysis. A distinct warbling signals the beginning of the analysis. The display shows which source is exposed and how much count time remains. On the back of the probe under the handle, an LED flashes to indicate that a source is exposed. Usually this LED flashes red. If it ever flashes yellow, replace the lithium battery in the probe (see *Chapter 5: Maintenance* for instructions). At the end of the analysis the Results Screen is displayed. If the automatic "store results" mode is enabled you will be prompted for a sample ID before the Results Screen is displayed.

Results Screen

RESULTS:		std. dev.
Cr22	4286	242
K	18498	266
Ca	100035	290
UP	DOWN	SEND STORE OPTS MEASR
1	2	3 4 5 6

UP or DOWN scrolls the screen to see more of the results. There are several actions you can select from the Results Screen (remember to press 0 to switch to PgUP and PgDN when you have lots of stored results).

SEND transmits the *result* report out the COM port. To send *spectra* out the COM port, select OPTS on the Results Screen. Then select Send Spectra.

STORE prompts you to enter an ID and then stores the results in memory. Memory is sufficient to store about 300 sets of results.

MEASR will immediately begin another analysis cycle. (CONT or the probe button will also start a new analysis at this point.)

OPTS There are two screens of special options under the Results Screen.

first screen

- | | | |
|---|-----------------------|---|
| 1 | Send spectra | Transmits all the spectra associated with the latest analysis out the COM port. |
| 2 | Store spectra | Prompts you for an ID and stores the spectra in memory. The unit can store approximately 40 three-spectrum sets (120 individual spectra). |
| 3 | Show std devs/units | Toggles the right column display between units of concentration and calculated standard deviation. |
| 4 | Show Application name | Displays the entire Application description while in the Application. |
| 5 | See raw data | Brings up a series of screens which allow you to inspect the x-ray data from several intermediate stages in the processing. This is mainly a diagnostic tool. |
| 6 | Autocycle | Initiates endless repetitive measurements. Primarily used for stability studies. (Press CONT to end autocycling.) |
| 7 | See results | Returns you to the Results Screen. |
| 8 | Rdy screen | Returns you to the Ready Screen. |
| 9 | More opts | Continues Results OPTS ... |
| 0 | Exit | Returns to Main Menu |

second screen

- | | | |
|---|------------------|--|
| 1 | Examine spectrum | Provides a variety of operations involving the display and manipulation of x-ray spectra on the screen. See <i>Spectrum Display</i> beginning on page 4-11 and/or flow chart on page 4-16 for details. |
|---|------------------|--|

2 Zero the current result

Zeros out the results of the last analysis and stores the corrections for use on all subsequent analyses within the current application. (Also, see Ready Screen—Options—3 Examine zero values.)

If zeroing has been performed, the message "zero" will appear on the Results Screen.

3 Enable/disable display thresholds

Normally very low analysis results are suppressed (not displayed) according to their values relative to the display thresholds. Disabling display thresholds shows all analysis results regardless of value (even negatives).

Modify

The Modify screen shows the count times currently set for each of the three sources and allows you to modify them. It also shows the x-ray energy range used for each of the three sources; this cannot be changed by the keypad.

SOURCE	RANGE(keV)		LIVE SECS		
>Cd109	33		200		
Fe55	8		200		
Am241	88		200		
UP	DOWN	MOD	REAL	EXIT	
1	2	3	4	5	6

UP and DOWN let you select the time you want to change. You also can change the Total Measurement Time. Existing time ratios are preserved (the Total Measurement Time you enter may be rounded off to keep the source times in whole seconds).

MOD allows you to enter a new count time value for a selected source. (CONT loads the new count time.)

REAL/LIVE toggles between real time (true clock time) and live time which adds time to the analysis to make up for the time the system is busy processing pulses.

EXIT tells the processor you are satisfied with the changes. The changes are saved and the Ready Screen is displayed.

Options

There are two screens of Ready Screen options.

first screen

- 1 **Energy Calibration** This is only occasionally necessary. Most Applications automatically perform an energy calibration with each analysis. A warning screen will inform you when an energy calibration is required. You will be prompted to place the safety cover on the probe. A 600 second analysis will start, at the end of which the software will automatically update the x-ray energy calibration.
- 2 **Acquire back-ground data** Prompts you to present blank samples so the back-ground subtraction coefficients can be updated. This procedure is only necessary when the unit exhibits a persistent zero drift on all blank samples.
- 3 **Examine zero values** Allows you to inspect (and modify) the current zero correction values. This procedure is only necessary when the unit exhibits a persistent zero drift on all blank samples.

If zeroing has been performed, "zero" will appear on the Results Screen.
- 4 **Show last results** Returns you to the Results Screen after performing other operations such as changing COM port parameters.
- 5 **Recall spectra and recalc** Lists the stored spectra-sets and allows you to select one for recalculation. Recalculation processes the stored spectra exactly as if they were just acquired from the probe.
- 6 **More options** Continues Options ...

second screen

- 1 Adjust calibration Lists all the elements in the application, with a slope and offset for each one. The default values of these coefficients are 1 and 0, respectively (in other words, no adjustment). If you want to adjust any element's calibration, enter the desired slope and offset here. The adjusted result (Ra) will be related to the original result (Ro) as follows:

$$Ra = (Ro * slope) + offset$$

Note: The sign of the offset is selected after its absolute value has been entered.

If a calibration adjustment has been made, "adj" will appear on the Results Screen.

- 2 Restore original calibration Restores all slope and offset calibration adjustments to 1 and 0.
- 3 Autocycle Causes the system to perform repeat analyses indefinitely. This feature is primarily used for studying the stability of the system. (Press CONT to end autocycling.)
- 4 Examine a spectrum Prompts you to select a spectrum, then displays it. See *Spectrum Display* (next page) for more information.
- 5 Enable/disable display thresholds Normally very low analysis results are suppressed (not displayed) according to their values relative to the display thresholds. Disabling display thresholds shows all of the analysis results regardless of value (even negatives).

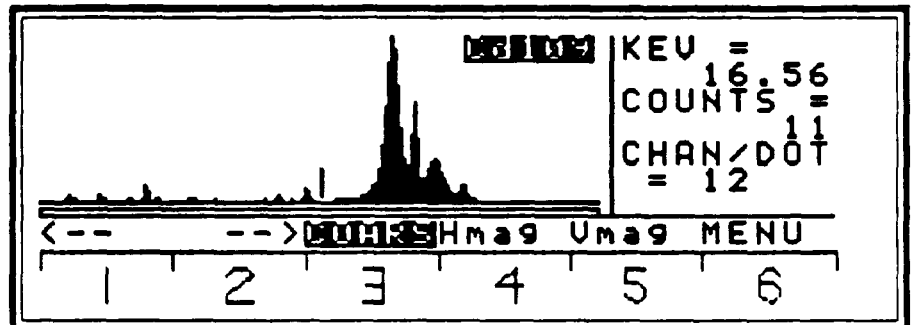
- 7 Return to Ready Screen

A flow chart of the Ready Screen Options is on page 4-17.

Spectrum Display

Several menu paths lead to Spectrum Display as illustrated in the flowcharts beginning on page 4-13. The most direct path (following a measurement) is through Results Screen—OPTS/second screen (as on page 4-7). A flow chart of this path begins on page 4-14.

When Spectrum Display is selected, you are asked to select one of three spectra (they are identified by source). With the spectrum displayed on the screen, there are a number of operations you can perform.



<--
-->

Move the cursor along the energy axis. The x-ray energy and the channel count corresponding to the cursor location are displayed on the right side of the screen.

COARS/FINE Toggle. Controls how fast the cursor moves.

Hmag Horizontal Magnification: Expands the display by a selectable factor centered at the cursor. The CHAN/DOT value will change according to the selected magnification.

Vmag Vertical Magnification: Provides

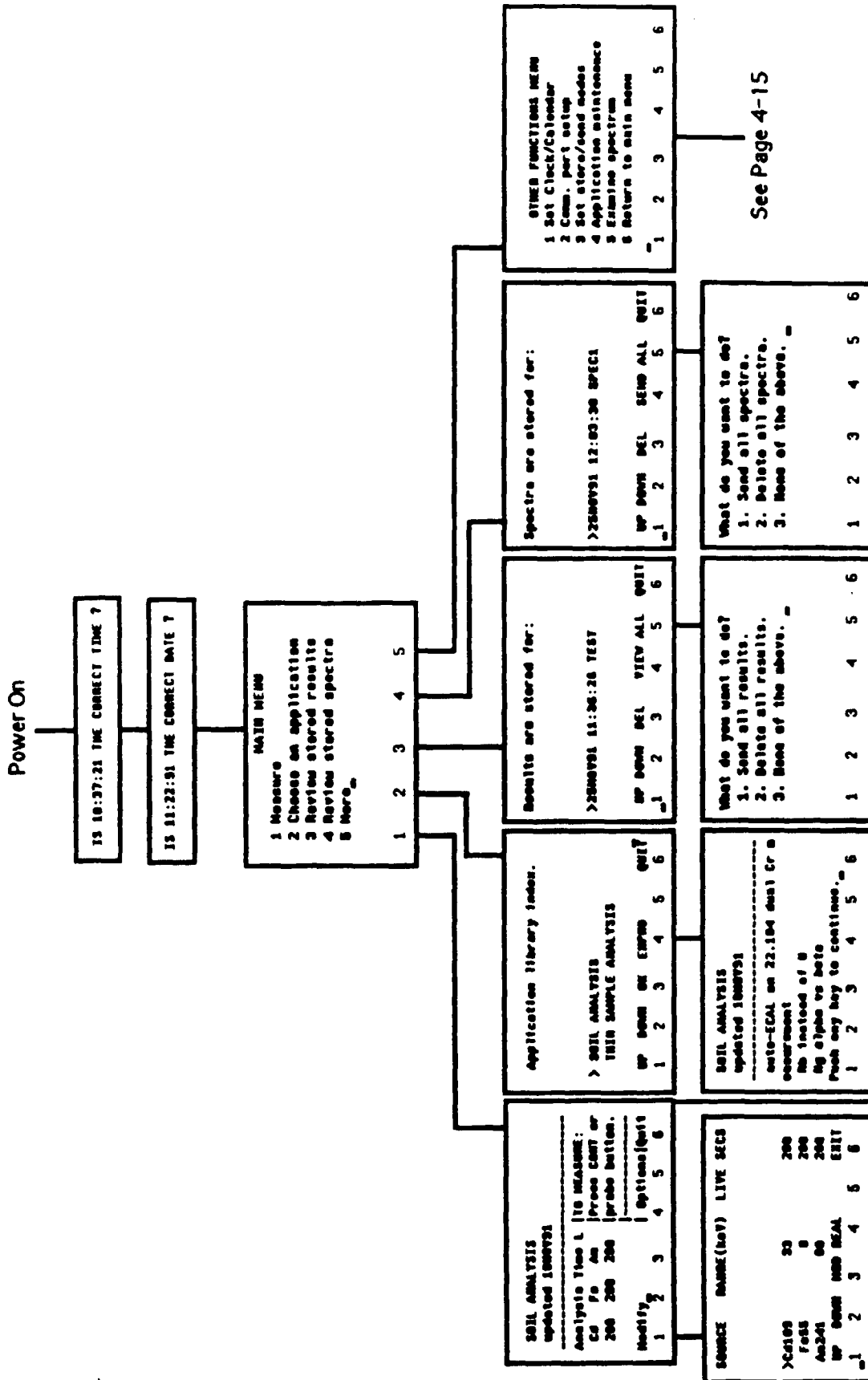
- manual adjustment of the vertical scale
- auto scaling so the highest peak is full scale
- logarithmic vertical scaling.

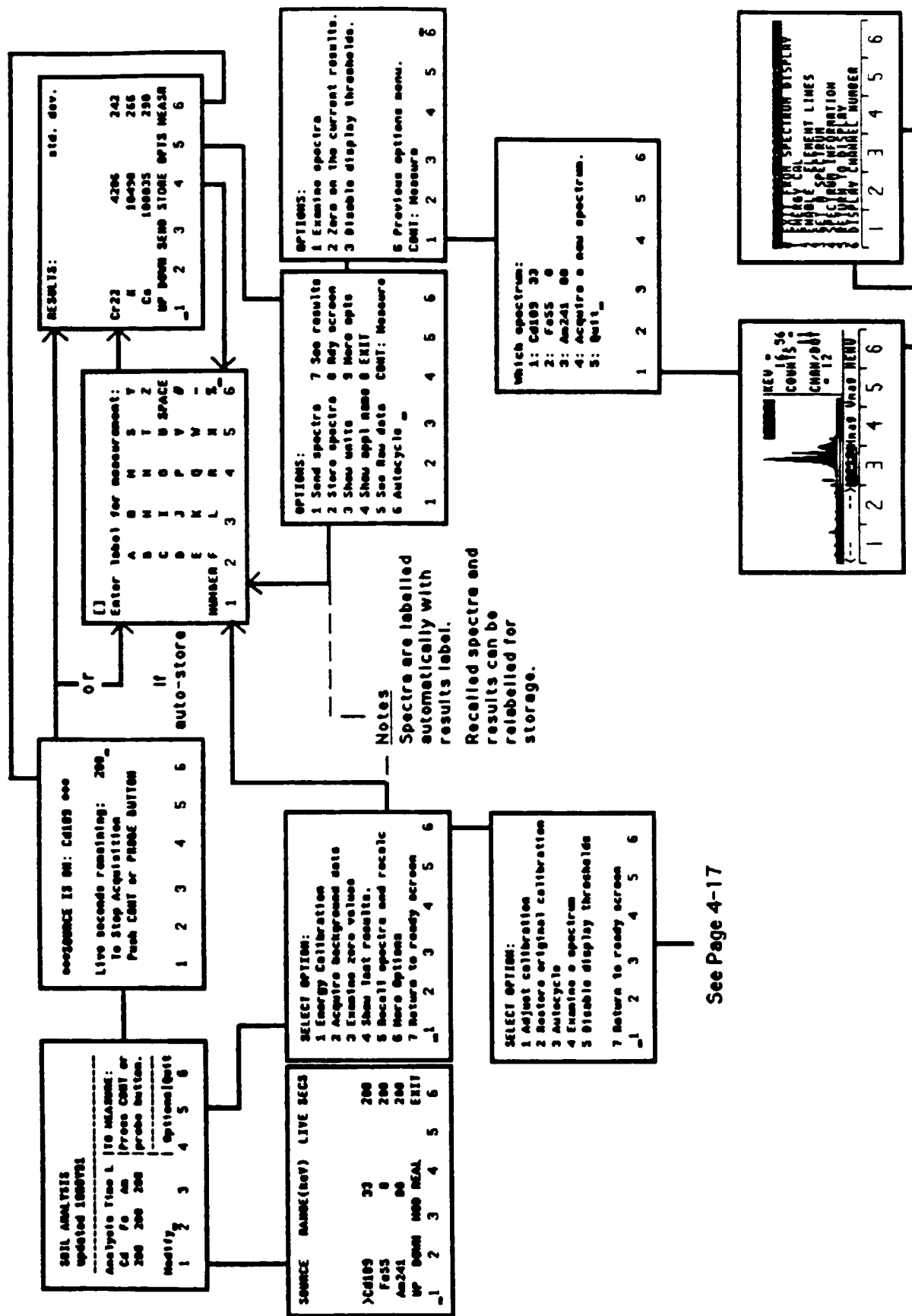
MENU Lists additional spectrum processing commands.

- | | |
|--------------------------------|--|
| 1 Energy cal | An advanced feature which allows you to perform an energy calibration by selecting known x-ray peaks and entering the energies for those peaks.

Note: This calibration will apply only to the current display. It is lost upon exiting the Spectrum Display mode. |
| 2 Enable/Disable element lines | Toggles the spectrum cursor function to display dotted vertical lines which indicate the expected location of the x-rays of a given element. The name of the element is displayed in the upper left corner. The <-- and --> keys move the lines either one element at a time or ten at a time, depending on the FINE/COARS selection. These lines are very useful for identifying unknown peaks in the spectrum. |
| 3 Get a spectrum | Lets you display another spectrum or acquire a new one. This feature is useful for rapid investigation of a series of unknown samples.

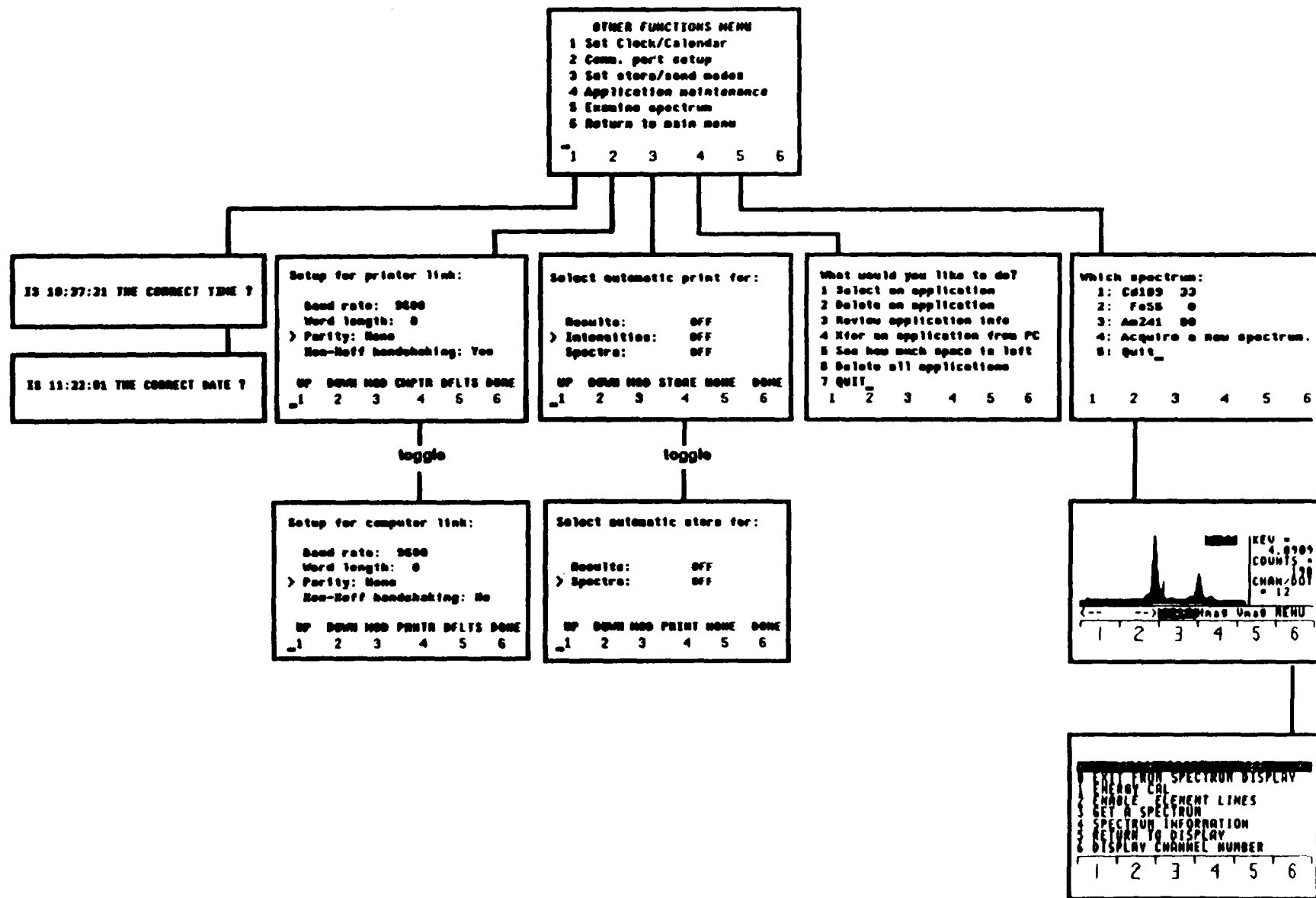
Note: Such acquired spectra carry the Energy Cal prescribed by the resident Application. If known peaks do not line up with the element lines, perform the above mentioned Energy Cal procedure. |
| 4 Spectrum Information | Not yet implemented. |
| 5 Return to Display | Returns you to Spectrum Display. |
| 6 Display Channel Number/keV | Toggles the cursor readout between energy units and channel number values. Used for diagnostic purposes. |



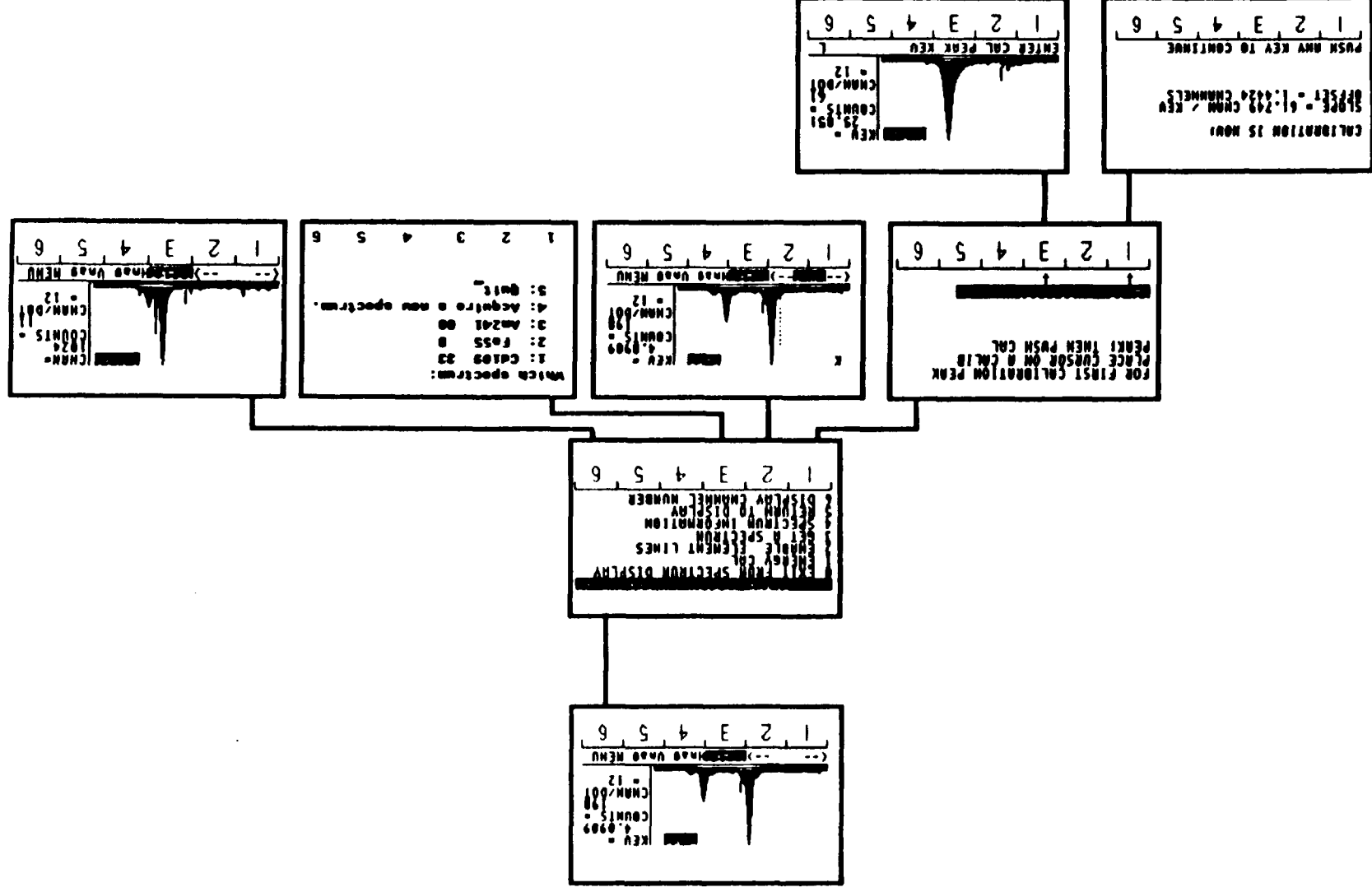


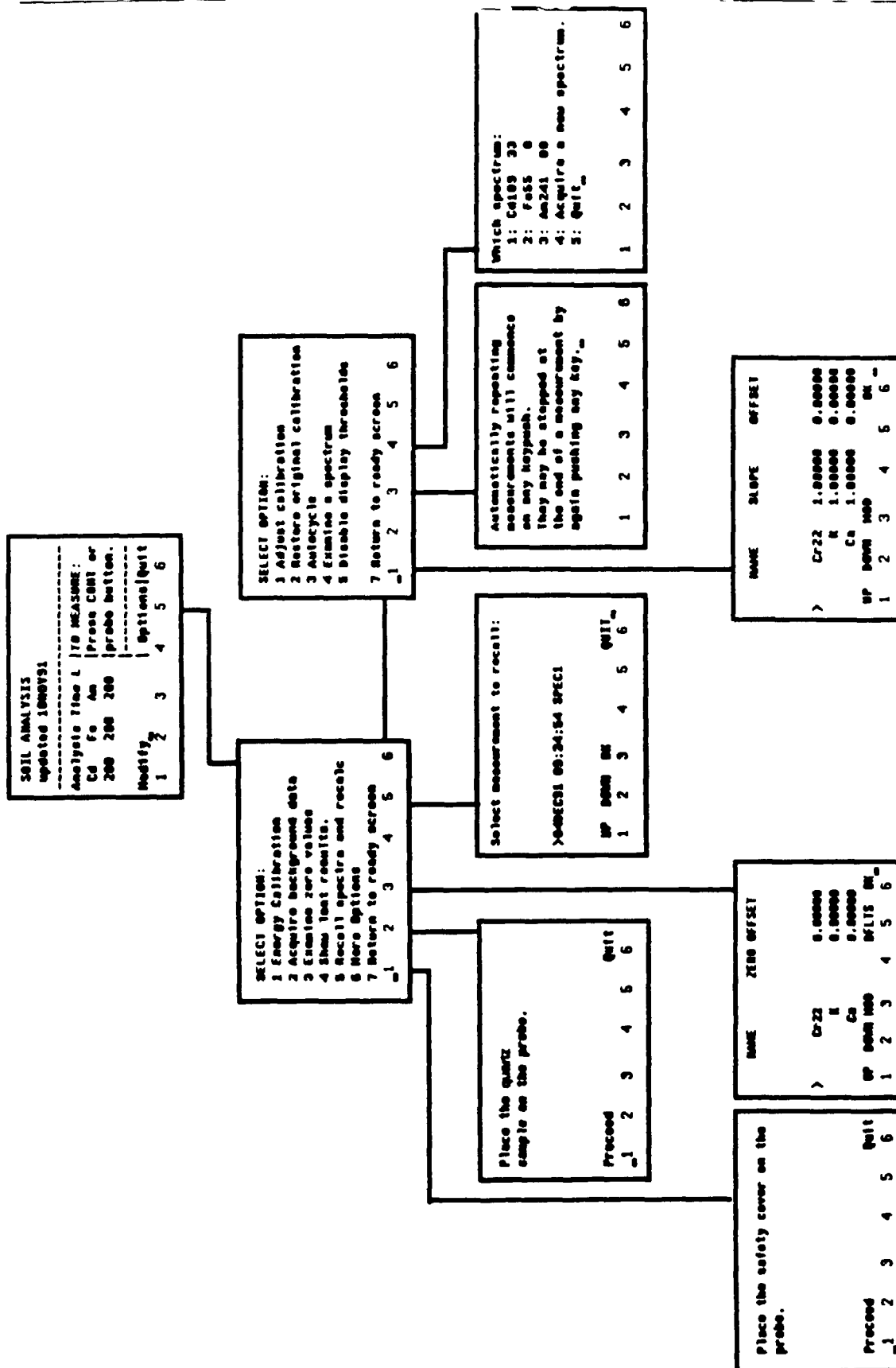
See Page 4-16

See Page 4-17



See Page 4-16





Chapter 5: Maintenance

Main Power Battery

The main power battery is a string of high-capacity rechargeable Ni-Cd cells. It will power the unit four to five hours at normal room temperatures (less time under very hot conditions). To charge the battery in the unit, the unit should be off so maximum power will be delivered to the battery. Once every two weeks the unit should be run without the charger connected until a Low Battery condition shuts the instrument down. Then, with the unit off, recharge the battery with a full 16-hour charge. This will restore the battery capacity. Auxiliary batteries *should not* be charged continuously. They should be given a 16-hour charge with the adapter and then set aside until needed.

Note: A nearly discharged battery may fail to turn the unit on even with the charger connected. If this happens you will need to allow up to 30 minutes for a sufficient charge or install a freshly charged battery. If your instrument is heavily used, you may want to purchase additional batteries and charging units.

Battery Replacement

When the battery will no longer hold a charge, it should be replaced. Only use TN part no. 885717; other Ni-Cds can reduce the performance of the instrument.

Probe Detector Bias Battery

The detector bias battery in the probe should be replaced every six months. In hot climates, or when the unit is often stored under conditions where the temperature exceeds 100°F (38°C), the battery may need to be replaced more often. Only use TN part no. 690352.

To ensure uninterrupted operation, your unit has been equipped with a sensing circuit on the bias battery. When the battery is near the end of its life, the red flashing SOURCE ON indicator (visible below the probe handle) will change to flashing yellow. Replace the battery immediately if this should happen.

Battery Replacement

1. Before starting, read these battery change instructions completely and make certain that the spare battery is at hand. Follow the order of the instructions *exactly* so that power to the bias supply is interrupted for as short a time as possible.
2. Set the probe face-down on the table in front of you. Orient it so the broad side is towards you and the handle is pointing off to the right.

3. Using a #1 Phillips screwdriver, remove the four screws holding the rear plate of the probe.
4. Carefully lift the rear edge of the rear plate (by lifting the handle) and tilt the plate back as if you were opening a box. It is necessary to tilt the plate as if it were on hinges because the wires at the back edge are fairly short. Tilt the plate past 90° and let it hang on the wires in the open position.
5. Look inside and, on the right, you will see the bias battery. It's a AA size lithium cell held in two spring clamps. It should be equipped with a pull-tab made of a wrap of fiberglass filament tape. If so, simply grasp the tab and pull straight up to remove the battery. If the tab breaks or is missing, insert and twist a large flat-blade screwdriver under the battery to pry it up out of the spring clamps.
6. Lift the battery up to the extent of its connecting wiring. This will expose the in-line connector. Pull the connector apart.
7. Immediately connect the new battery, being careful not to bend the connector pins. Perform this step quickly to minimize the interruption in the detector bias.
8. Lower the new battery into place making sure that its wires lie comfortably in the space provided and are not caught in the spring clamps. Orient the new battery so the wire that runs along the battery (under the battery wrapping) is upwards. Press the new battery into the spring clamps.
9. Raise the rear plate to 90° and hold it up so there is no tension on the wires. Press on the red wire connectors to make sure they are fully seated. They often are pulled away by this operation and must be reseated for proper operation.
10. Slowly lower the rear plate towards the closed position, tucking in any wires that try to escape. Press the rear plate firmly into position. It should move relatively easily down to a point where the gasket is actually touching the enclosure. If modest hand pressure cannot close this gap, lift the rear plate again and check for wires in the way. Never force the rear plate down into place.
11. Once the rear plate is properly reseated, replace the four screws and tighten securely.

Probe Window Replacement

If the probe window is punctured it must be replaced immediately. If not replaced, the probe and internal mechanisms of the unit can be contaminated. This can lead to costly repairs. Replacement windows are supplied in the accessory case and additional supplies can be ordered as TN part No. 885868.

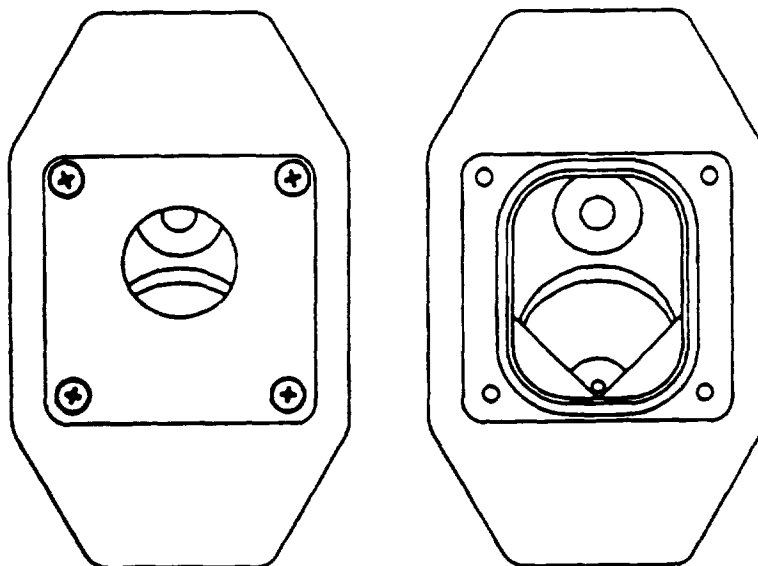
Caution: *The detector window is extremely fragile. During the window replacement operation take care to keep pointed tools and loose objects well away from the open area of the probe.*

1. Place the probe in the lab stand.
2. Note the location of the window aperture; it is closer to one end of the window plate.
3. Remove the four screws from the window plate. Do not remove any other screws. Be careful to not drop the screws inside the probe (this could happen if the probe window is badly punctured).

Probe Window Aperture

With Window Plate

Without Window Plate



4. Press any corner of the window plate. The opposite corner will pop up so that you can easily lift out the window. Rock the window plate gently if it does not immediately pop up.

5. The O-ring may pop out of place. To put it back, stretch it for 10 seconds and lay it back in the groove. The O-ring must lie flat in the groove in order to install the new window plate.
6. Put in the new window assembly in the same orientation as the old. Check that the screw holes line up exactly. If the surface of the window plate is not flush with the face of the probe, the O-ring has probably come out of the groove. Remove the window assembly and try again.
7. Carefully replace the four screws.

Chapter 6: Radiation Safety

Warning: All operators should read, understand, and observe the following instructions to minimize any radiation exposure risk.

The Spectrace 9000 is designed to be portable and easy to use, even for operators with no prior experience of x-ray analysis. It is important to realize that x-rays are used in measurement and that radioisotope sources of x-rays are contained in the probe. We have designed into the instrument many special features to minimize the risk of accidental radiation exposure, but there are still precautions which you must take to ensure safe operation.

Routine Precautions

- Familiarize yourself with basic instrument operation before attempting analysis.
- Do not initiate an analysis unless the probe is connected to the electronics and a sample is in place.
- Do not remove a sample or move the probe about while the indicators show SOURCE ON.

SOURCE ON indicators are:

- the message on the screen
....SOURCE ON....
- the flashing light at the base of the probe.
- Avoid contact with the top of the probe.
- When using sample cups, use the lab stand shield.
- Never aim the probe at yourself or another person.
- Always replace a damaged probe window as soon as possible (see *Chapter 5: Maintenance* for replacement instructions).
- Never attempt to loosen any of the screws on the probe, except those associated with the replacement of the probe window or the battery unit in the base.
- In case of serious mechanical damage to the probe, return the complete instrument to TN Technologies for inspection.

In Case of Malfunction

As long as the analysis sequence is properly carried out and a sample is in contact with the probe window, there is no radiation hazard.

Warning: With a malfunction, a source may be exposed. Follow the instructions below to avoid any radiation exposure risk.

With any malfunction, your first priority must be to keep the probe aperture covered.

Software Failure

If the processor ever locks up during a measurement (the keyboard will be inoperative and the screen may go blank):

1. Disconnect the probe from the data processor unit. This will automatically close the shutter.
2. To restore proper operation of the processor, press the OFF key, wait a few moments and press the ON key. This should bring up a display.

If it does not, remove the main battery for a moment, then reconnect it. By doing so, you may lose data.

3. Turn the data processor OFF again before reconnecting the probe.
-

Hardware Failure

The opening and closing of a source is sensed by the electronics through a switch contact. When the program terminates the exposure, it looks to the switch contact to verify the closing of the source. If the switch contact does not detect that the source has closed, the following message will appear:

*** WARNING ***
Source did not close

Should you ever see this message, follow these steps:

1. Cover the probe end with the safety shield. Place the probe in the carrying case or other secure place.
 2. Call TN Technologies immediately at (512) 388-9200. ^{9/00} The instrument must not be used in this condition.
 3. Do not leave the instrument unattended.
-

Problem Prevention

Always take appropriate steps to keep the instrument operating reliably. This will reduce the chance of malfunction or of any situation which might become hazardous. Make sure the probe is placed securely on the sample or in the lab stand before beginning an analysis. If the instrument is used routinely on the bench with the probe in the lab stand, use the shield provided with the lab stand. Avoid immersing the probe.

Report any instrument malfunction to TN Technologies, no matter how infrequent or trivial it may seem.

Safety Provisions in the Instrument Design

We have designed the Spectrace 9000 with many special features to minimize the risk of accidental radiation exposure. These design features are outlined below:

- All radioactive materials are permanently sealed within high strength capsules which have undergone tests to be certified as satisfactory for industrial use.
- The source capsules are firmly mounted within the instrument, and are further protected against physical damage by the probe design.
- When sources are not exposed (any time other than during a measurement) all radiation emitted by the sources is stopped by the shielding structure.
- Only one source at a time can be exposed, and the operator is alerted to an exposed source by visible and audible signals.
- Source exposure is only possible under electrical input from the electronic unit, and then only at certain points in the application procedure.
- Accidental source exposure is *not* possible. The correct key sequence must be entered before a measurement can be started.
- The radiation field from each source is spatially restricted, and in normal use is totally confined by the sample to the immediate vicinity of the probe.

Source	Max. Activity	Half-Life
Iron 55 (Fe-55)	100 millicuries	2.7 years
Cadmium 109 (Cd-109)	10 millicuries	1.3 years
Americium 241 (Am-241)	10 millicuries	458 years
Curium 244 (Cm-244)	100 millicuries	17.6 years

The source capsules are located beneath and to one side of the measurement aperture, and are enclosed in source holders mounted on a rotatable hub. Radiation is emitted through the aperture when the hub is rotated to expose one source at a time to the sample. The activity level and radiation output of these sources require that the probe always be handled with care. In normal instrument operation there is no radiation hazard.

Two of the primary sources have comparatively short half-lives. As each source decays, the remaining activity decreases by 1%, approximately every week for Cd-109 and every two weeks for Fe-55. Measurement times are typically increased in proportion to source decay. In terms of potential radiation exposure, the need for caution remains the same.

Source Capsule Design

The radioactive material of each source (except Fe-55) is sealed inside a stainless steel capsule with a beryllium window. This capsule is of standard industrial design and tested integrity. The Fe-55 source is electroplated onto a metal substrate and then overplated with nickel to seal it.

The sources are further protected and additional shielding is provided by metal source holders. The source holders are attached to a hub which is rotated by an electric motor to expose one source at a time through the measurement aperture. The hub has a narrow clearance between its top surface and the lower surface of the probe cover, which is also shielded. The effect of the design is to absorb all of the source radiation inside the top region of the probe when the sources are in the closed position. A fail safe circuit in the probe monitors power levels from the electronics, and in the event of a power failure, uses energy stored in a capacitor to drive the sources to the safe position.

The integrity of the source capsules has been tested according to standards recommended for industrial radioisotope x-ray devices (ANSI Standard N542 "Classification of Sealed Radioactive Sources").

Radiation Levels

When the source shutters are closed, the external radiation level is very low. With three sources, Fe-55, Cd-109 and Am-241 at their maximum activity and the source mechanism in the "closed" position, the gamma and x-ray exposure rate is <0.1 mR/h. When a 100 mCi Cm-244 source is loaded, the neutrons emitted due to spontaneous fission create a 5 mrem/h exposure rate at a 5 cm (1.95") distance from the source. At 12", the rate drops to 0.14 mrem/h. When the source shutters are open, external radiation levels remain low, provided the window is covered by a full size sample in contact with the probe. If the window is only partially covered, exercise caution to prevent unnecessary exposure. When using the probe in the lab stand, use the shield to prevent unnecessary exposure.

Warning: With no sample in place, the radiation level is not negligible, especially in the measurement zone close to the window. The instrument should never be operated without a sample in place on the window.

Chapter 7: Regulatory Requirements

Licensing

Instruments containing quantities of radioactive material, except those specifically exempted, are subject to regulations by the U. S. Nuclear Regulatory Commission (NRC) or an Agreement State. An "Agreement State" is one which has entered into an agreement with the NRC, in which regulatory authority over the use of certain materials within the state has been transferred to the respective state. These agreements provide for a high degree of compatibility between the regulatory programs of the state and the NRC. Because of the similarities between the programs of the Agreement States and the NRC, we can discuss some general characteristics of all programs as they pertain to instrument control.

The approach utilized in assuring the safety of a manufactured device containing radioactive material is to regulate the manufacturer. The inherent safety of the device is studied, and conditions of assuring its safety are contained in the specific license under which the device is manufactured and distributed. The user must also be licensed either by a Specific License or General License prior to receiving the device. Most users accept the 9290 Analyzer (Spectrace 9000) under General License which is issued in all regulations. General License conditions are summarized on the label affixed to the probe unit. This label must not be removed. Complete General License conditions are included in this manual.

Leak Testing

The sources contained in the 9290 Analyzer are required to be leak tested at intervals not to exceed six (6) months. TN Technologies will automatically notify you when leak tests are due, and can provide a mailable leak test kit. The analyzer must be within current leak test prior to any shipment.

Transportation

Under U. S. Department of Transportation regulations (49 CFR, 173.422) and International Air Transport Association (IATA), the 9290 Analyzer is classified as "Radioactive material, excepted package, instruments, UN2910." As such, the device can be transported by any mode - air, land, or sea. It is eligible to be transported in the baggage compartment of a passenger-carrying aircraft. The device is excepted from all specification packaging, marking, and labeling (i.e. no marking or labeling is required on the carrying case). The bill of lading or airway bill should, however, contain the words: "Radioactive material, excepted package, instruments, UN2910." These words should be inserted with the description of the goods. Since regulations are constantly changing, call TN Technologies (512/388-9291) for current information prior to making a shipment.

Additional Safety Items

In the event of an accident which is thought to have damaged the source encapsulations, TN Technologies should be notified immediately for further instructions.

Until appropriate measurements have been made, the device should be handled as little as possible to avoid the spread of contamination. TN Technologies will provide advice and help in the evaluation of further steps to be taken.

Should use of the analyzer be discontinued, or disposal of the sources become necessary, TN Technologies should be contacted for aid in disposition. Disposal of radioactive material must be handled by someone specifically licensed to do so.

Appendix: General Licenses

IMPORTANT

PLEASE READ

**EXCERPTS FROM TEXAS REGULATIONS FOR CONTROL OF
RADIATION AS THEY APPLY TO ANY PERSON WHO RECEIVES,
ACQUIRES, POSSESSES OR USES RADIOACTIVE MATERIAL
RECEIVED UNDER GENERAL LICENSE**

**TN Technologies, Inc.
Post Office Box 800
Round Rock, Texas 78680**

January, 1992

To our Valued Customers:

You recently received device(s) containing radioactive material distributed under General License as set out in the regulations. TN Technologies, Inc. (formerly Texas Nuclear Corporation), as the manufacturer and distributor, is required to furnish parts of the Texas regulations pertaining to General Licensees. Identical wording is contained in all Agreement State and Nuclear Regulatory Commission regulations.

The attached excerpts outline your responsibilities as a licensee and the conditions under which you must possess, use and transfer these devices. Regulations can be confusing to read and interpret; therefore, following is a brief summary of those items of which you should be most aware.

41.22; 41.31; 41.40; 11.4

- (1) Only certain devices are eligible for distribution to General Licensees. This approval is granted to the manufacturer under a special license.
- (2) Fixed gauges must be commissioned by the manufacturer or someone holding a specific license authorizing them to perform these functions. Gauges can be physically installed (mounted) without a specific license authorization. Also, wipe test samples can be taken for return to an approved laboratory, and periodic shutter checks can be performed by operating personnel.
- (3) Ensure leak testing is performed at specified intervals. For Texas Nuclear products, this is:

Gauges	Every 3 years
Portable Analyzers	Every 6 months
- (4) Maintain records as follows:

Receipt records
Leak test certificates
Copy of General License Regulations
Records of transfer or disposal
Records of periodic shutter (on-off mechanism) tests
- (5) Devices can only be transferred to someone authorized to receive them.

41.50

- (1) The General License can be amended in the regulations.
- (2) Willful violation of license conditions can result in termination of license by the regulatory agency.

41.100

- (1) If you need to ship radioactive material, contact TN for instructions.
-

11.5

- (1) The agency may inspect any time and the licensee must make records available.

11.6: 11.7

- (1) Devices must be leak tested at specified intervals and records maintained.
- (2) Test results must not exceed 0.005 μCi .

21.402: 21.403

- (1) You must report to the regulatory agency any lost radioactive material and certain incidents involving radioactive material.
- (2) Reportable incidents are stated in 21.403.

Please feel free to call if you have any questions.

Doris C. Bryan, Manager
Licensing & Regulatory Affairs
(512) 388-9287

41.22 General Licenses - Radioactive Material other than Source Material**(d) Certain Measuring, Gauging and Controlling Devices**

- (1) A general license is hereby issued to commercial and industrial firms and to research, educational, and medical institutions, individuals in the conduct of their business, and State or local government agencies to receive, acquire, possess, use, or transfer in accordance with the provisions of 41.22(d)(2), (3), (4), radioactive material, excluding special nuclear material, contained in devices designed and manufactured for the purpose of detecting, measuring, gauging or controlling thickness, density, level, interface location, radiation, leakage, or qualitative or quantitative chemical composition, or for producing light or an ionized atmosphere.
- (2) The general license in 41.22(d)(1) applies only to radioactive material contained in devices which have been manufactured and labeled in accordance with the specifications contained in a specific license issued by the U. S. Nuclear Regulatory Commission or an Agreement State which authorizes distribution of devices to persons generally licensed by the U.S. Nuclear Regulatory Commission or an Agreement State.
- (3) Any person who receives, acquires, possesses, uses, or transfers radioactive material in a device pursuant to the general license in 41.22(d)(1):
 - (i) Shall assure that all labels affixed to the device at the time of receipt, and bearing a statement that removal of the label is prohibited, are maintained thereon and shall comply with all instructions and precautions provided by such labels;
 - (ii) Shall ensure that the device is tested for leakage of radioactive material and proper operation of the on-off mechanism and indicator, if any, at no longer than six-month intervals or at such other intervals as specified in the label; however,
 - (a) Devices containing only krypton need not be tested for leakage of radioactive material, and
 - (b) Devices containing only tritium or not more than 100 microcuries of other beta and/or gamma emitting material or 10 microcuries of alpha emitting material and devices held in storage in the original shipping container prior to initial installation need not be tested for any purpose;
 - (iii) Shall assure that the tests required by 41.22(d)(3)(ii) and other testing, installation, servicing, and removal from installation involving the radioactive materials, shielding or containment, are performed by a person holding a specific license from the Agency, the U.S. Nuclear Regulatory Commission, or an Agreement State to perform such activities;

-
- (iv) Shall maintain records showing compliance with the requirements of 41.22(d)(3)(ii) and (3)(iii). The records shall show the results of tests. The records also shall show the dates of performance of, and the names of persons performing, testing, installation servicing, and removal from installation concerning the radioactive material, its shielding or containment;
 - (v) Upon occurrence of a failure of or damage to, or any indication of a possible failure of or damage to, the shielding of the radioactive material, shall immediately suspend the operation of the device until it has been repaired by the manufacturer or other person holding a specific license from the Agency, the U.S. Nuclear Regulatory Commission, or Agreement State to repair such devices, or disposed of by transfer to a person authorized by a specific license to receive the radioactive material contained in the device and, within thirty (30) days, furnish to the Agency a report containing a brief description of the event and the remedial action taken;
 - (vi) Shall not abandon the device containing radioactive material;
 - (vii) Except as provided in 41.22(d)(3)(viii), shall transfer or dispose of the device containing radioactive material only by transfer to a specific licensee of the Agency, the U.S. Nuclear Regulatory Commission, or an Agreement State whose specific license authorizes him to receive the device and within thirty (30) days after transfer of a device to a specific licensee shall furnish to the Agency a report containing identification of the device by manufacturer's name and address of the person receiving the device. No report is required if the device is transferred to the specific licensee in order to obtain a replacement device;
 - (viii) Shall transfer the device to another general licensee only:
 - (a) Where the device remains in use at a particular location. In such case the transferor shall give the transferee a copy of this regulation and any safety documents identified in the label on the device and within thirty (30) days of the transfer, report to the Agency the manufacture's name and model number of device transferred, the name and address of the transferee, and the name and/or position of an individual who may constitute a point of contact between the Agency and the transferee; or
 - (b) Where the device is held in storage in the original shipping container at its intended location of use prior to initial use by a general licensee.
 - (ix) Shall comply with the provisions of 21.402 and 21.403 for reporting radiation incidents, theft, or loss of licensed material, but shall be exempt from the other requirements of Parts 21 and 22.
- (4) The general license in 41.22(d)(1) does not authorize the manufacture of devices containing radioactive material.
 - (5) The general license provided in 41.22(d)(1) is subject to the provisions of Parts 11, 41.31, 41.40, 41.50 and 41.100.

- (a) Each license issued pursuant to this part shall be subject to all the provisions of the Act, now or hereafter in effect, and to all rules, regulations, and orders of the Agency.
- (b) No license issued or granted under this part and no right to possess or utilize radioactive material granted by any license issued pursuant to this part shall be transferred, assigned, or in any manner disposed of, either voluntarily or involuntarily, directly or indirectly, through transfer of control of any license to any person unless the Agency shall, after securing full information, find that the transfer is in accordance with the provisions of the Act, and shall give its consent in writing.
- (c) Each person licensed by the Agency pursuant to this part shall confine his use and possession of the material licensed to the locations and purposes authorized in the license.
- (d) (1) Each licensee shall notify the Agency, in writing, immediately following the filing of a voluntary or involuntary petition for bankruptcy under any Chapters of Title 11 (Bankruptcy) of the United States Code (11 U.S.C.) by or against:
 - (i) A licensee;
 - (ii) An entity [as that term is defined in 11 U.S.C. 101(14)] controlling a licensee or listing the license or licensee as property of the estate; or
 - (iii) An affiliate [as the term is defined in 11 U.S.C. 101(2)] of the licensee.
- (2) This notification must indicate:
 - (i) The bankruptcy court in which the petition for bankruptcy was filed; and
 - (ii) The date of the filing of the petition.

- (a) No licensee shall transfer radioactive material except as authorized pursuant to this part.
- (b) Except as otherwise provided in his license and subject to the provisions of 41.40(c) and (d), any licensee may transfer radioactive material:
 - (1) To the Agency; 11/
 - (2) To the U.S. Energy Research and Development Administration;
 - (3) To any person exempt from the regulations in this part to the extent permitted under such exemption;

11/ A licensee may transfer material to the Agency only after receiving prior approval from the Agency.

-
- (4) To any person authorized to receive such material under terms of a general license or its equivalent, or a specific license or equivalent licensing document, issued by the Agency, the U.S. Nuclear Regulatory Commission, or any Agreement State, or to any person otherwise authorized to receive such material by the Federal Government or any agency thereof, the Agency, or any Agreement State; or
- (5) As otherwise authorized by the Agency in writing.
- (c) Before transferring radioactive material to a specific licensee of the Agency, the U.S. Nuclear Regulatory Commission, or an Agreement State, or to a general licensee who is required to register with the Agency, the U.S. Nuclear Regulatory Commission, or an Agreement State prior to receipt of the radioactive material, the licensee transferring the material shall verify that the transferee's license authorizes the receipt of the type, form, and quantity of radioactive material to be transferred.
- (d) The following methods for the verification required by 41.40(c) are acceptable:
- (1) The transferor may have in his possession, and read, a current copy of the transferee's specific license or registration certificate;
 - (2) The transferor may have in his possession a written certification by the transferee that he is authorized by the license or registration certificate to receive the type, form, and quantity of radioactive material to be transferred, specifying the license or registration certificate number, issuing agency, and expiration date;
 - (3) For emergency shipments the transferor may accept oral certification by the transferee that he is authorized by license or registration certificate to receive the type, form, and quantity of radioactive material to be transferred, specifying the license or registration certificate number, issuing agency, and expiration date; provided, that the oral certification is confirmed in writing within ten (10) days.
 - (4) The transferor may obtain other sources of information compiled by reporting service from official records of the Agency, the U.S. Nuclear Regulatory Commission, or the licensing agency of an Agreement State as to the identity of licensees and the scope and expiration dates of licenses and registration; or
 - (5) When none of the methods of verification described in 41.40(d)(1) to (4) are readily available or when a transferor desires to verify that information received by one of such methods is correct or up-to-date, the transferor may obtain and record information from the Agency, the U.S. Nuclear Regulatory Commission, or the licensing agency of the Agreement State that the transferee is licensed to receive the radioactive material.
- (e) Preparation for shipment and transport of radioactive material shall be in accordance with the provisions of 41.100.

41.50 Modification, Revocation, and Termination of Licenses

- (a) The terms and conditions of all licenses shall be subject to amendment, revision, or modification, or the license may be suspended or revoked by reason of amendments of the Act, or by reason of rules, regulations, and orders issued by the Agency.
- (b) Any license may be revoked, suspended, or modified, in whole or in part, for any material false statement in the application or any statement of fact required under provisions of the Act, or because of conditions revealed by such application or statement of fact or any report, record, or inspection, or other means which would warrant the Agency to refuse to grant a license on an original application, or for violations of, or failure to observe any of the terms and conditions of the Act, or of the license, or of any rule, regulation, or order of the Agency.
- (c) Except in cases of willfulness or those in which the public health, interest or safety requires otherwise, no license shall be modified, suspended, or revoked unless, prior to the institution of proceedings therefor, facts or conduct which may warrant such action shall have been called to the attention of the licensee in writing and the licensee shall have been accorded an opportunity to demonstrate or achieve compliance with all lawful requirements.

41.100 Preparation of Radioactive Material for Transport

No licensee shall deliver any radioactive material to a carrier 12/ for transport unless:

- (a) The licensee complies with the applicable requirements of the regulations, appropriate to the mode of transport, of the U.S. Department of Transportation insofar as such regulations relate to the packing of radioactive material, and to the monitoring, marking, and labeling of those packages;
- (b) The licensee has established procedures for opening and closing packages in which radioactive material is transported to provide safety and to assure that, prior to the delivery to a carrier for transport, each package is properly closed for transport; and
- (c) Prior to delivery of a package to a carrier for transport, the licensee shall assure that any special instructions needed to safely open the package are sent to, or have been made available to the consignee.

11.4 Records

Each licensee and registrant shall maintain records showing the receipt, transfer, and disposal of all licensed or registered sources of radiation. These records shall be maintained by the licensee or registrant until disposal is authorized by the Agency. Additional record requirements are specified elsewhere in these rules. All records required by these rules shall be accurate and factual.

12/ For the purpose of the regulation, a licensee who transports his own licensed material as a private carrier is considered to have delivered such material to a carrier for transport.

11.5 Inspections

- (a) Each licensee and registrant shall afford the Agency at all reasonable times opportunity to inspect sources of radiation and the premises and facilities wherein such sources of radiation are used or stored.
- (b) Each licensee and registrant shall make available to the Agency for inspection, upon reasonable notice, records maintained pursuant to these rules.
- (c) **Routine Inspection of Radiation Machines and Services**
 - (1) Routine inspections by Agency personnel will be made no more frequently than the intervals specified in Appendix 11-C.
 - (2) Notwithstanding the provisions of 11.5(c)(1) above, for those radiation machines determined by the Agency to constitute a minimal threat to human health and safety, the routine inspection interval will be five years. The applicable categories are listed in Appendix 11-D.
 - (3) Notwithstanding the inspection intervals specified in these rules, the Agency may inspect registrants more frequently due to:
 - (i) The persistence or severity of violations found during an inspection.
 - (ii) Investigation of an incident or complaint concerning the facility;
 - (iii) A request for an inspection by a worker(s) in accordance with 22.16;
 - (iv) Any change in a facility or equipment which might cause a significant increase in radiation output or hazard; or
 - (v) A mutual agreement between the Agency and registrant.

11.6 Tests

Each licensee and registrant shall perform, upon instructions from the Agency, or shall permit the Agency to perform such reasonable tests as the Agency deems appropriate or necessary including, but not limited to, tests of:

- (a) Sources of radiation;
- (b) Facilities wherein sources of radiation are used or stored;
- (c) Radiation detection and monitoring instruments; and
- (d) Other equipment and devices used in connection with utilization or storage of licensed or registered sources of radiation.

11.7 Tests for Leakage and/or Contamination of Sealed Sources

- (a) **Requirements.** Each licensee using sealed sources of radioactive material, Nickel 63 foil sources, or plated alpha sources shall have such sources periodically tested for leakage and/or contamination as prescribed in this Section. Records of these tests shall be maintained and made available for inspection by the Agency.
- (b) **Method of Testing.** Tests for leakage and contamination shall be performed only by persons specifically authorized to perform such tests by the Agency, another Agreement State or the U.S. Nuclear Regulatory Commission. The test sample shall be taken from the surface of the source or from the surface of the device in which the source is stored or mounted and on which one might expect contamination to accumulate. The test sample shall be analyzed for radioactive contamination and the analysis shall be capable of detecting the presence of 0.005 microcuries of radioactivity on the test sample. The results of the test shall be kept in units of microcuries and maintained for inspection by the Agency.
- (c) **Interval of Testing.** Each sealed source of radioactive material and each Nickel 63 foil source shall be tested at intervals not to exceed six (6) months except that each source designed for the purpose of emitting alpha particles shall be tested at intervals not to exceed three (3) months. In the absence of a certificate from a transferor indicating that a test has been made prior to the transfer, the sealed source shall not be put into use until tested. Notwithstanding the leak test intervals specified herein, the Agency may authorize extended leak test intervals for specified sources used in certain specific applications.
- (d) **Leaking or Contaminated Sources.** If the test reveals the presence of 0.05 microcuries or more of leakage or contamination obtained from a teletherapy or a gamma irradiator source, or 0.005 microcuries or more of leakage or contamination obtained from any other type source, the licensee shall immediately withdraw the source from use and shall cause it to be decontaminated, repaired, or disposed of in accordance with the Texas Regulations for Control of Radiation. A report, describing the equipment involved, the test results and the corrective action taken shall be filed with:

Director, Division of Compliance and Inspection
Bureau of Radiation Control
Texas Department of Health
1100 West 49th Street
Austin, TX 78756

- (e) **Exemptions.** Notwithstanding the requirements of 11.7(a) through (d), the following sources are exempted from periodic leak testing:
 - (1) Hydrogen 3 sources;
 - (2) Sources of radioactive material with a half-life of 30 days or less;
 - (3) Sealed sources of radioactive material in gaseous form;
 - (4) Sources of beta and/or gamma emitting radioactive material with an activity of 100 microcuries or less;
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- (5) Sources of alpha emitting radioactive material with an activity of 10 microcuries or less;
 - (6) Nickel 63 foil sources of 100 microcuries or less; and
 - (7) Plated alpha sources, other than Californium 252 sources, with an activity of 0.1 microcuries or less.

21.402 Reports of Theft or Loss of Sources of Radiation

- (a) Each licensee or registrant shall report by telephone and telegraph, mailgram, or facsimile, to the Agency the theft or loss of any licensed or registered source of radiation immediately after such occurrence becomes known.
- (b) Each licensee or registrant who is required to make a report pursuant to 21.402(a) shall, within thirty (30) days after he learns of the loss or theft, make a report in writing to the Agency, setting forth the following information:
 - (1) A description of the source of radiation involved, including the kind, quantity, chemical, physical form, and/or model and serial numbers;
 - (2) A description of the circumstances under which the loss or theft occurred;
 - (3) A statement of disposition or probable disposition of the source of radiation involved;
 - (4) Radiation exposures to individuals, circumstances under which the exposures occurred, and the extent of possible hazard to persons in unrestricted areas;
 - (5) Actions which have been taken, or will be taken, to recover the source of radiation, and
 - (6) Procedures or measures which have been or will be adopted to prevent a recurrence of the loss or theft of sources of radiation.
- (c) Subsequent to filing the written report, the licensee or registrant shall also report any substantive additional information on the loss or theft which becomes available to the licensee, within thirty (30) days after he learns of such information.

21.403 Notification of Incidents

- (a) **Immediate Notification.** Each licensee or registrant shall immediately notify the Agency by telephone and telegraph, mailgram, or facsimile of any incident involving any source of radiation possessed by him and which may have caused or threatens to cause:
 - (1) A dose to the whole body of any individual of 25 rems or more of radiation; a dose to the skin of the whole body of any individual of 150 rems or more of radiation, or a dose to the feet, ankles, hands, or forearms of any individual of 375 rems or more of radiation; or
 - (2) The release of radioactive material in the concentrations which, if averaged over a period of twenty-four (24) hours, would exceed 5,000 times the limits specified for such materials in Appendix A, Table II; or

- (3) A loss of one working week or more of the operation of any facilities affected; or
 - (4) Damage to property in excess of \$200,000.
- (b) Twenty-Four Hour Notification. Each licensee or registrant shall within twenty-four (24) hours notify the Agency by telephone and telegraph, mailgram, or facsimile, of any incident involving any source of radiation possessed by him and which may have caused or threatens to cause:
- (1) A dose to the whole body of any individual of five (5) rems or more of radiation; a dose to the skin of the whole body of any individual of thirty (30) rems or more of radiation; or a dose to the feet, ankles, hands, or forearms of seventy-five (75) rems or more of radiation; or
 - (2) The release of radioactive material in concentrations which, if averaged over a period of twenty-four (24) hours, would exceed 500 times the limits specified for such materials in Appendix A, Table II; or
 - (3) A loss of one day or more of the operation of any facilities affected; or
 - (4) Damage to property in excess of \$2,000.

LICENSING AGENCIES IN AGREEMENT STATES AS OF JANUARY, 1992

ALABAMA

Alabama Dept. of Public Health
Div. of Radiological Health
State Office Building
Montgomery, AL 36130
Phone: 205/242-5315

ARIZONA

Arizona Radiation Regulatory Agency
814 South 40th Street
Phoenix, AZ 85040
Phone: 602/255-4845

ARKANSAS

Arkansas Dept. of Health
Radiation Control & EMP
4815 West Markham Street
Little Rock, AR 72205-3867
Phone: 501/661-2301

CALIFORNIA

California Department of Health
Radiologic Health Section
744 P Street
Sacramento, CA 95814
Phone: 916/445-0931

COLORADO

Colorado Department of Health
Radiation Control Division
4210 East 11th Avenue
Denver, CO 80220
Phone: 303/331-8480

FLORIDA

Florida Department of HRS
Radiological Health Services
1317 Winewood Boulevard
Building 1, Room 108
Tallahassee, FL 32301
Phone: 904/487-2437

GEORGIA

Georgia Department of
Natural Resources
Radioactive Materials Program
4244 International Parkway, Suite 114
Atlanta, GA 30354
Phone: 404/362-2675

ILLINOIS

Illinois Department of Nuclear Safety
1035 Outer Park Drive
Springfield, IL 62704
Phone: 217/785-9948

IOWA

Iowa Department of Public Health
Bureau of Radiological Health
Lucas State Office Building
Des Moines, IA 50319
Phone: 515/281-4942

KANSAS

Kansas Department of Health
and Environment
Bureau of Radiation Control
Topeka, KS 66620
Phone: 913/296-1562

KENTUCKY

Radiation Control Branch
Cabinet for Human Resources
275 East Main Street
Frankfort, KY 40621
Phone: 502/564-3700

LOUISIANA

Louisiana Department of
Environmental Quality
Radiation Protection Division
P. O. Box 82135
Baton Rouge, LA 70884-2135
Phone: 504/765-0160

LICENSING AGENCIES IN AGREEMENT STATES AS OF JANUARY, 1992

MARYLAND

Department of the Environment
2500 Broening Highway
Baltimore, MD 21224
Phone: 301/631-3000

MISSISSIPPI

Mississippi State Board of Health
Div. of Radiological Health
P. O. Box 1700
Jackson, MS 39215-1700
Phone: 601/960-7400

NEBRASKA

Nebraska Department of Health
Div. of Radiological Health
P. O. Box 95007
Lincoln, NE 68509
Phone: 402/471-2168

NEVADA

Nevada Department of Human Resources
Bureau of Regulatory Health
Consumer Health Protection
505 East King Street
Carson City, NV 89710
Phone: 702/687-5394

NEW HAMPSHIRE

New Hampshire Division of
Public Health Services
Bureau of Radiological Health
Health & Welfare Building
6 Hazen Drive
Concord, NH 03301-6527
Phone: 603/271-4588

NEW MEXICO

New Mexico Environmental
Improvement Division
Community Services Bureau
1190 St. Francis Drive
Santa Fe, NM 87503
Phone: 505/827-2948

NEW YORK

New York Department of Labor
Div. of Safety and Health
1 Main Street, 8th Floor
Brooklyn, NY 11201
Phone: 718/797-7636

NORTH CAROLINA

North Carolina Department of
Environment, Health and Natural
Resources
Division of Radiation Protection
Radioactive Materials Section
P. O. Box 27687
Raleigh, NC 27611-7687
Phone: 919/571-4141

NORTH DAKOTA

North Dakota Department of Health
Environmental Health Section,
Environmental Engineering
1200 Missouri Avenue, Room 304
Bismarck, ND 58502
Phone: 701/221/5183

OREGON

Oregon Department of Human Resources
Health Division
Radiation Control Section
1400 SW 5th Avenue
P. O. Box 231
Portland, OR 97201
Phone: 503/229-5797

LICENSING AGENCIES IN AGREEMENT STATES AS OF JANUARY, 1992

RHODE ISLAND

Rhode Island Radiation
Control Agency
206 Cannon Building
75 Davis Street
Providence, RI 02908
Phone: 401/277-2438

UTAH

Utah Department of Health
Division of Environmental Health
Bureau of Radiation Control
P. O. Box 1660
1460 West 288 North
Salt Lake City, UT 84116-0690
Phone: 801/538-6734

SOUTH CAROLINA

South Carolina Dept. of Health
and Environmental Control
Div. of Radioactive Material
Licensing and Compliance
2600 Bull Street
Columbia, SC 29201
Phone: 803/734-4626

WASHINGTON

Radioactive Materials Section
DOH - Division of Radiation
Protection LE-13
Airdustrial Center Building #5
P. O. Box 47827
Olympia, WA 98504-7827
Phone: 206/753-3468

TENNESSEE

Tennessee Department of Public Health
Division of Radiological Health
TERRA Building
150 9th Avenue North
Nashville, TN 37203
Phone: 615/741-7812

TEXAS

Texas Department of Health
Bureau of Radiation Control
Division of Licensing,
Registration, and Standards
1100 West 49th Street
Austin, TX 78756
Phone: 512/835-7000

LICENSING AGENCIES IN AGREEMENT STATES AS OF JANUARY, 1992**HEADQUARTERS OFFICE**

Division of Fuel Cycle
and Material Safety
Office of Nuclear Safety
and Safeguards
U.S. Nuclear Regulatory Commission
Washington, DC 20555
Phone: 301/492-7000

**IDAHO, MONTANA, OKLAHOMA,
SOUTH DAKOTA, WYOMING**

U.S. Nuclear Regulatory Commission
Region IV
Material Radiation Protection Section
611 Ryan Plaza Drive
Suite 1000
Arlington, TX 76011
Phone: 817/860-8100

**CONNECTICUT, DELAWARE, MAINE
MASSACHUSETTS, NEW JERSEY
PENNSYLVANIA, VERMONT, WASHINGTON, DC**

U.S. Nuclear Regulatory Commission
Region I
Nuclear Material Section B
475 Allendale Road
King of Prussia, PA 19406
Phone: 215/337-5000

ALASKA, HAWAII, GUAM

U.S. Nuclear Regulatory Commission
Region V
Material Radiation Protection Section
1450 Maria Lane, Suite 210
Walnut Creek, CA 94596
Phone: 510/975-0200

**VIRGINIA, WEST VIRGINIA,
PUERTO RICO, VIRGIN ISLANDS**

U.S. Nuclear Regulatory Commission
Region II
Material Radiation Protection Section
101 Marietta Street, Suite 2900
Atlanta, GA 30303
Phone: 404/331-4503

**INDIANA, MICHIGAN, MINNESOTA,
MISSOURI, OHIO, WISCONSIN**

U.S. Nuclear Regulatory Commission
Region III
Material Licensing Section
799 Roosevelt Road
Glen Ellyn, IL 60137
Phone: 708/790-5500

ENVIRONMENTAL AND TECHNICAL SERVICES

James B. (Jim) Whitworth
Director
(512) 388-9285

Doris C. Bryan
Manager
(512) 388-9287

Contacts:

Consulting - Safety Programs	Doris Bryan	(512) 388-9287
Emergencies/Accidents	Mark McCray Doris Bryan	(512) 388-9301 (512) 388-9287
Leak Test Service	Sharon Alexander	(512) 388-9303
Disposals	Mark McCray	(512) 388-9301
Licensing and Regulatory Affairs	Doris Bryan Kathleen Nelson	(512) 388-9287 (512) 388-9288
Training	Pam Zelewski	(512) 388-9286
Shipping Radioactive Material	Doris Bryan	(512) 388-9287
Survey Meter Calibration & Repair	Jon George	(512) 388-9308

Glossary

Accuracy	A measure of how close an analysis result is to the correct value or to a value assumed to be the correct value.												
Application	A complete analysis configuration which defines the elements to be measured, the sources to be used, the interfering elements in the sample, and a set of fundamental parameter calibration coefficients.												
Backscatter	The source radiation that "scatters" off the sample, losing little or no energy in the process.												
Blank	A sample that contains <i>none</i> of the elements of interest. Used to zero the unit.												
Channels	One of 2048 memory locations the x-ray spectral data are stored in after being processed by a multi-channel analyzer.												
Fundamental Parameters	Theoretical XRF calibration coefficients based on known mass absorption coefficients, fluorescent yields, etc. for all elements.												
keV	Kilo-electron-volt. Unit of energy associated with x-ray emission.												
Precision	A measure of the repeatability of an analysis result on the same sample under identical measurement conditions.												
Source Decay	<p>The gradual reduction in source activity over time. The "half-life" of the source is the time it takes for the source to decay to 1/2 its original activity. The Spectrace 9000's sources and their half-lives are:</p> <table><tr><td>isotope</td><td>half-life</td><td>%decay/week</td></tr><tr><td>Fe-55</td><td>2.7 years</td><td>.5</td></tr><tr><td>Cd-109</td><td>1.3 years</td><td>1</td></tr><tr><td>Am-241</td><td>456 years</td><td>.003</td></tr></table>	isotope	half-life	%decay/week	Fe-55	2.7 years	.5	Cd-109	1.3 years	1	Am-241	456 years	.003
isotope	half-life	%decay/week											
Fe-55	2.7 years	.5											
Cd-109	1.3 years	1											
Am-241	456 years	.003											
Spectrum	The distribution of x-ray energies emitted by a sample when excited by a source.												
Standard Deviation	<p>A quantitative measure of the typical error observed (or expected) in an analysis result. Specifically, in a group of results obtained by repeat analyses of the same sample, 67% of the results will fall within one standard deviation of the mean result. Ninety five percent of the results will fall within two standard deviations of the mean. The term "sigma" is synonymous with "standard deviation."</p>												

EXHIBIT III

STANDARD METHOD D-2216-80, MOISTURE CONTENT OF SOIL



Standard Method for Laboratory Determination of Water (Moisture) Content of Soil, Rock, and Soil-Aggregate Mixtures¹

This standard is issued under the fixed designation D 2216; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This method covers the laboratory determination of the water (moisture) content of soil, rock, and soil-aggregate mixtures by weight. For simplicity, the word "material" hereinafter refers to either soil, rock, or soil-aggregate mixtures, whichever is most applicable.

1.2 The water content of a material is defined as the ratio, expressed as a percentage, of the mass of "pore" or "free" water in a given mass of material to the mass of the solid material particles.

1.3 This method does not give true representative results for materials containing significant amounts of halloysite, montmorillonite, or gypsum minerals; highly organic soils; or materials in which the pore water contains dissolved solids (such as salt in the case of marine deposits). For a material of the previously mentioned types, a modified method of testing or data calculation may be established to give results consistent with the purpose of the test.

2. Summary of Method

2.1 The practical application in determining the water content of a material is to determine the mass of water removed by drying the moist material (test specimen) to a constant mass in a drying oven controlled at $110 \pm 5^\circ\text{C}$ and to use this value as the mass of water in the test specimen. The mass of material remaining after oven-drying is used as the mass of the solid particles.

3. Significance and Use

3.1 For many soil types, the water content is one of the most significant index properties used in establishing a correlation between soil behavior and an index property.

3.2 The water content of a soil is used in almost every equation expressing the phase relationships of air, water, and solids in a given volume of material.

3.3 In fine-grained (cohesive) soils, the consistency of a given soil type depends on its water content. The water content of a soil, along with its liquid and plastic limit, is used to express its relative consistency or liquidity index.

3.4 The term "water" as used in geotechnical engineering, is typically assumed to be "pore" or "free" water and not that which is hydrated to the mineral surfaces. Therefore, the water content of materials containing significant amounts of

hydrated water at in-situ temperatures or less than 110°C can be misleading.

3.5 The term "solid particles" as used in geotechnical engineering, is typically assumed to mean naturally occurring mineral particles that are not readily soluble in water. Therefore, the water content of materials containing extraneous matter (such as cement, etc), water-soluble matter (such as salt) and highly organic matter typically require special treatment or a qualified definition of water content.

4. Apparatus

4.1 *Drying Oven*, thermostatically-controlled, preferably of the forced-draft type, and maintaining a uniform temperature of $110 \pm 5^\circ\text{C}$ throughout the drying chamber.

4.2 *Balances*, having a precision (repeatability) of ± 0.01 g for specimens having a mass of 200 g or less; ± 0.1 g for specimens having a mass of between 200 and 1000 g, or ± 1 g for specimens having a mass greater than 1000 g.

4.3 *Specimen Containers*—Suitable containers made of material resistant to corrosion and a change in mass upon repeated heating, cooling, and cleaning. Containers with close-fitting lids shall be used for testing specimens having a mass of less than about 200 g; while for specimens having a mass greater than about 200 g, containers without lids may be used (Note 1). One container is needed for each water content determination.

NOTE 1—The purpose of close-fitting lids is to prevent loss of moisture from specimens before initial weighing and to prevent absorption of moisture from the atmosphere following drying and before final weighing.

4.4 *Desiccator*—A desiccator of suitable size (a convenient size is 200 to 250-mm diameter) containing a hydrous silica gel. This equipment is only recommended for use when containers having close-fitting lids are not used. See 7.4.1.

5. Samples

5.1 Keep the samples that are stored prior to testing in noncorrodible airtight containers at a temperature between approximately 3 and 30°C and in an area that prevents direct contact with sunlight.

5.2 The water content determination should be done as soon as practicable after sampling, especially if potentially corrodible containers (such as steel thin-walled tubes, paint cans, etc.) or sample bags are used.

6. Test Specimen

6.1 For water contents being determined in conjunction with another ASTM method, the method of specimen selection specified in that method controls.

¹ This method is under the jurisdiction of ASTM Committee D-18 on Soil and Rocks and is the direct responsibility of Subcommittee D18.03 on Texture, Density, and Density Characteristics of Soils.
Current edition approved May 30, 1980. Published July 1980. Originally published as D 2216 - 63 T. Last previous edition D 2216 - 71.

6.2 The manner in which the test specimen is selected and dried mass is basically dependent on the purpose (application) of the test, type of material being tested, and the size of sample (specimen from another test, bag, tube, pit-barrel, etc.). In all cases, however, a representative portion of the total sample shall be selected. If a layered soil more than one soil type is encountered, select an average portion or individual portions or both, and note which portion(s) was tested in the report of the results.

6.2.1 For bulk samples, select the test specimen from the material after it has been thoroughly mixed. The mass of moist material selected shall be in accordance with the following table:

Sieve Retaining More Than About 10 % of Sample	Recommended Minimum Mass of Moist Specimen, g
2.0 mm (No. 10) sieve	100 to 250
4.75 mm (No. 4) sieve	300 to 500
19 mm	500 to 1000
38 mm	1500 to 3000
76 mm	5000 to 10 000

6.2.2 For small (jar) samples, select a representative portion in accordance with the following procedure:

6.2.2.1 For cohesionless soils, thoroughly mix the material, then select a test specimen having a mass of moist material in accordance with the table in 6.2.1. See Note 2.

6.2.2.2 For cohesive soils, remove about 3 mm of material from the exposed periphery of the sample and slice it in half to check if the material is layered) prior to selecting the test specimen. If the soil is layered see 6.2. The mass of moist material selected should not be less than 25 g or should be in accordance with the table in 6.2.1 if coarse-grained particles are noted. (Note 2).

6.3 Using a test specimen smaller than the minimum mass indicated previously requires discretion, though it may be adequate for the purpose of the test. A specimen having a mass less than the previously indicated value shall be noted in the report of the results.

NOTE 2—In many cases, when working with a small sample containing a relatively large coarse-grained particle, it is appropriate not to include this particle in the test specimen. If this occurs, it should be noted in the report of the results.

7. Procedure

7.1 Select representative test specimens in accordance with Section 6.

7.2 Place the moist specimen in a clean, dry container of known mass (Note 3), set the lid securely in position, and determine the mass of the container and moist material using an appropriate balance (4.2). Record these values.

7.3 Remove the lid and place the container with moist material in a drying oven maintained at $110 \pm 5^\circ\text{C}$ and dry to a constant mass (Notes 4, 5, and 6).

NOTE 3—To assist in the oven-drying of large test specimens, they should be placed in containers having a large surface area (such as pans) and the material broken up into smaller aggregations.

NOTE 4—The time required to obtain constant mass will vary depending on the type of material, size of specimen, oven type and capacity, and other factors. The influence of these factors generally can be established by good judgment, and experience with the materials being tested and the apparatus being used. In most cases, drying a test specimen over night (about 16 h) is sufficient. In cases where there is doubt concerning the adequacy of drying, drying should be continued until the mass after two successive periods (greater than $\frac{1}{2}$ h) of drying

indicate an insignificant change (less than about 0.1 %). Specimens of sand may often be dried to constant mass in a period of about 4 h, when a forced-draft oven is used.

NOTE 5—Oven-drying at $110 \pm 5^\circ\text{C}$ does not always result in water content values related to the intended use or the basic definition especially for materials containing gypsum or other minerals having significant amounts of hydrated water or for soil containing a significant amount of organic material. In many cases, and depending on the amount of organic material, it might be more applicable to intended use for these types of materials, it might be more applicable to maintain the drying oven at $60 \pm 5^\circ\text{C}$ or use a vacuum desiccator at a vacuum of approximately 133 Pa (10 mm Hg) and at a temperature ranging between 23 and 60°C for drying. If either of these drying methods are used, it should be noted in the report of the results.

NOTE 6—Since some dry materials may absorb moisture from moist specimens, dried specimens should be removed before placing moist specimens in the oven. However, this requirement is not applicable if the previously dried specimens will remain in the drying oven for an additional time period of about 16 h.

7.4 After the material has dried to constant mass remove the container from the oven and replace the lid. Allow the material and container to cool to room temperature or until the container can be handled comfortably with bare hands and the operation of the balance will not be affected by convection currents. Determine the mass of the container and oven-dried material using the same balance as used in 7.2. Record this value.

7.4.1 If the container does not have a lid, weigh the container and material right after their temperatures are such that the operation of the balance will not be affected by convection currents or after cooling in a desiccator.

NOTE 7—Cooling in a desiccator is recommended since it prevents absorption of moisture from the atmosphere during cooling.

8. Calculation

8.1 Calculate the water content of the material as follows:

$$w = [(W_1 - W_2)/(W_2 - W_c)] \times 100 = \frac{W_w}{W_s} \times 100$$

where:

w = water content, %;
 W_1 = mass of container and moist specimen, g;
 W_2 = mass of container and oven-dried specimen, g;
 W_c = mass of container, g;
 W_w = mass of water, g, and
 W_s = mass of solid particles, g.

9. Report

9.1 The report (data sheet) shall include the following:

9.1.1 Identification of the sample (material) being tested by boring number, sample number, test number, etc.

9.1.2 Water content of the specimen to the nearest 0.1 % or 1 %, depending on the purpose of the test.

9.1.3 Indication of test specimen having a mass less than the minimum indicated in Section 6.

9.1.4 Indication of test specimen containing more than one soil type (layered, etc).

9.1.5 Indication of the method of drying if different from oven-drying at $110 \pm 5^\circ\text{C}$.

9.1.6 Indication of any material (size and amount) excluded from the test specimen.

10. Precision and Accuracy

10.1 Requirements for the precision and accuracy of this test method have not yet been developed.

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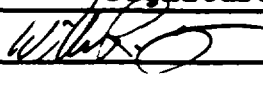
EXHIBIT IV
STANDARD OPERATING PROCEDURES

OHM Corporation

**QUALITY POLICY AND
PROCEDURE APPROVAL
AND REVISION RECORD**

Document # QP-613	Title Pile Sampling
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APPROVAL

Name	Title	Signature	Date
W. Moody	Supn.-Plans/Procs.		27 NOV 95
	APPROVED FOR USE.		
	FU management approval pending.		

REVISION RECORD

Ltr.	Date	Change Description	Initials							
A		issue	-	-	-	-	-	-	-	-
B										
C										
D										
E										
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STANDARD OPERATING PROCEDURE

Title: Pile Sampling

Document #: QP-613

Date Issued:

Rev. 02

Date: 12 OCT 93

1.0 PURPOSE

The purpose of this procedure is as follows:

- 1.0 Describe the requirements for collecting soil pile samples.
- 2.0 Define the methods for finding soil pile volumes.

2.0 SCOPE

The following information outlines the general procedures for collecting samples from soil piles and other waste material piles, equipment necessary for sampling, and the collection of a representative sample of the material. Also presented will be factors for consideration when formulating a sampling plan.

3.0 RELATED DOCUMENTS

3.1 Surface Soil Sampling SOP, OHM QP-609

3.2 ASTM E-300, Standard Practice for Sampling Industrial Chemicals

3.3 Preparation of Soil Sampling Protocol: Techniques and Strategies, (EPA 600/4-83-020), Dr. Benjamin J. Mason, Prepared under contract to the USEPA, Environmental Monitoring Systems Laboratory-Las Vegas, August 1983

3.4 Soil Sampling Quality Assurance User's Guide, (EPA 600/4-84-043), Dr. Delbert S. Barth and Dr. Benjamin J. Mason, Prepared by the Environmental Research Center, University of Nevada, Las Vegas under a cooperative agreement with the USEPA, May 1984

4.0 GENERAL INFORMATION

Soils are often non-homogeneous and distribution of contaminants in a soil is often non-uniform. To offset these during sampling work the following are accomplished.

- 1.0 Collect samples with high volume-to-surface area ratios.
- 2.0 Use a systematic (grid pattern) approach to sample collection.
- 3.0 Maintain a detailed record during sampling operations, particularly noting location, depth, and characteristics such as grain size, color and odor, and/or readings obtained on field monitoring equipment.

Soil sampling equipment needs to meet the following general requirements.

- 1.0 Materials of construction shall be stainless steel, plastic or teflon coated steel. Chrome or nickel plated steel should not be used. Brass sampling tools can be used if they are non-reactive and non-contaminating to the sample medium.
- 2.0 Materials of construction shall be compatible with and non-reactive to compounds of interest.
- 3.0 Materials of construction will not leave residues in samples that will interfere with analyses of samples.

Soil bioactivity and problems with volatilization of organic compounds from the soil require that the following be accomplished for soil samples immediately after collection.

- 1.0 Tightly seal containers.
- 2.0 Refrigerate samples to $\sim 4^{\circ}\text{C}$.
- 3.0 Remove samples from exposure to sunlight.

4.1 Pile Sampling QA/QC

Quality control samples will be taken at the discretion of the client and OHM management. Provisions for quality control sampling are usually included in the CSAP.

Types of Quality Control samples that might be taken during a pile sampling operation can include Matrix Spike/Matrix Spike Duplicates, Field Spikes, Duplicates, and/or Split Samples.

4.2 Pile Sampling Precautions

1. In order to collect samples that accurately represent levels of contamination found in the pile, a grid will usually need to be developed. This may require the use of mathematical formulas, a calculator, and various measurement techniques to determine the height, and length and width of the pile.
2. Other methods of sampling a pile are to divide the pile into sections and grid sample each section, or to take a set number of samples for every (x) number of cubic yards of soil (i.e. ten samples for every 500 yd³ of soil in the pile).
3. The problems associated with developing a grid to sample a pile can be eliminated by obtaining a sample from every (y) number of buckets that are placed onto the pile (i.e. one 8oz sample collected from every 3 Trackhoe buckets emptied into the pile). These samples can be stored on ice and composited daily. When excavation is completed there should be a good representation of the concentrations of soil in the pile. However, compositing of samples is **NOT** to be done if analyses for volatile organic compounds will be performed on the samples.

4. Soil piles may be physically unstable. Precautions may need to be taken so that sampling can be safely accomplished. This in effect may limit the ways in which a pile can be sampled.

4.3 Preparation for Sampling

Whenever a pile sampling project is performed, it is always necessary to obtain measurements of the pile(s) which are used for calculating the volume of the pile(s). Two methods are used for pile volume calculations. One method is for conical or pyramidal piles and the other is for elongated or prism shaped piles.

4.3.1 Volume of Conical or Pyramidal Pile

Step 1. Measure height and base parameters (i.e. length/width for rectangle, radius of circle, etc.) of pile.

Step 2. Determine area of base of pile. If base of pile is irregularly shaped divide into regularly shaped areas and figure separately. Add areas together to obtain total area of base.

Square/Rectangle:	$A = L \cdot W$
Circle:	$A = \pi \cdot r^2$
Ellipse:	$A = \pi \cdot a \cdot b$
Triangle:	$A = \frac{1}{2} B \cdot h$

Where:

A = Area of the base of the pile
L = Length of one side of a square or rectangle
W = Width of other side of rectangle
 π = 22/7 or 3.14
r = radius of circle
a = $\frac{1}{2}$ of semiminor axis
b = $\frac{1}{2}$ of semimajor axis
B = Base length of triangle
h = Distance from base to top of triangle perpendicular to base.

Step 3. Determine volume of pile. For all base shapes use the following equation:

$$V = \frac{1}{2} A \cdot H$$

Where:

V = Volume of pile
A = Area of the base of the pile
H = Height to apex of the pile

NOTE: If the top of the pile is not complete, estimate height to apex from top of pile. Determine volume of pile as if it were complete to estimated apex. Determine volume of missing top part in the same manner. Subtract missing top from estimated total pile volume to obtain actual pile volume.

4.3.2 Volume of an Elongated or Prism Shaped Pile

Step 1. Divide pile into uniform shaped (along length) body and sides. Measure pile accordingly (length of body, height, dimensions of sides).

Step 2. Determine the area of the face of the body by defining it's shape and then using the formula for that shape.

Triangular Area = $\frac{1}{2} B \cdot h$
Semicircular Area = $\frac{1}{2} \pi \cdot r^2$

Where:

B = Base of triangle, width of pile
h = Height of triangle/pile
 π = 22/7 or 3.14
r = Radius of circle, height of pile

Step 3. Determine volume of the body of the pile. For all face shapes use:

$$V = A \cdot L$$

Where:

- V = The volume of the body of the pile**
- A = The area of the face of the body**
- L = The length of the body**

Step 4. Determine the volume of the sides of the pile.

The sides of the pile can be treated as conical shaped piles and their volumes determined as such. The volumes of the sides can then be added to the volume of the body of the pile to find the total volume of the pile.

$$TV = VB + VS1 + VS2$$

Where:

- TV = Total volume of pile**
- VB = Volume of body of pile**
- VS1 = Volume of one side of pile**
- VS2 = Volume of other side of pile**

4.3.3 Volume of Irregularly Shaped Piles

Many piles will not exhibit dimensions exactly the same as those described above. These piles should be divided into more regularly shaped sections and their volumes determined independently. These separated volumes can then be added to obtain the total volume of the pile.

5.0 DEFINITIONS

- 5.1 Environmental Sample - low concentration sample typically collected off site and not requiring DOT hazardous waste labelling as a high hazard sample.
- 5.2 Hazardous Waste Sample - medium to high concentration sample (e.g., source material, sludge leachate) requiring DOT labelling and Contract Lab handling as a high hazard sample.
- 5.3 Ellipse - a closed symmetrical figure bounded by a curve like an elongated circle.
 - 5.3.1 Semimajor axis - a line segment from the center of the ellipse to its boundary along the longest dimensions.
 - 5.3.2 Semiminor axis - a line segment from the center of the ellipse to the its boundary along the dimension perpendicular to the longest one.
- 5.4 Regularly shaped areas - all those areas which shape can be approximated by the following: rectangle, circle, triangle and ellipse.

6.0 RESPONSIBILITIES

6.1 Field Operations Leader

The Field Operations Leader is responsible for the overall safety of the sampling operations. This includes informing and obtaining help from local authorities if necessary, selection of sample points, and halt of operations if necessary.

6.2 Sampling Technicians

The Sampling Technicians are responsible for the following.

- 1.0 Reading and implementing project sampling plans.
- 2.0 Accumulating and dispatching appropriate supplies and equipment to accomplish the sampling objectives.
- 3.0 Locating and marking sampling points.

- 4.0 Decontamination of field sampling gear in a correct and appropriate manner consistent with site requirements.
- 5.0 Collecting, preserving and packaging soil pile samples.
- 6.0 Maintaining sampling records, sampling logs, data sheets and maps.
- 7.0 Filling-out Chain-of-Custody forms and appropriate log books.
- 8.0 Maintaining physical custody of samples until they are transferred off-site or to an on-site laboratory.

7.0 PROCEDURES

7.1 Sampling Procedures

As with soil sampling, soil or waste pile samples can be collected at the surface or at depth, and different equipment will be required in each instance. Surface samples can be collected most efficiently with a trowel or scoop. For samples at depth, a decontaminated bucket auger may be required to advance the hole, then another decontaminated auger used for sample collection. For a sample core, waste pile samplers or grain samplers may be used.

7.1.1 Surface Soil Samples

- 1.0 Locate the sample point and clear away surface debris. Mark the sampling point with a surveyor's stake if possible.
- 2.0 Collect an adequate volume of soil or waste material from a depth of 0-6 inches using a shovel to initially open up the pile surface and then using a trowel or scoop to obtain the actual sample material.
- 3.0 Immediately transfer the material to a sample container and seal the container.

7.1.2 Surface Soil Core Samples

- 1.0 For a core sample from the surface proceed as in 1.0 and 2.0 above and then use a waste pile sampler, trier or tube corer to obtain a sample in place of using the trowel.
- 2.0 Transfer the sample directly into the sample container, or use a decontaminated trowel, tongue depressor or spatula for transfer if necessary. A wide mouth bottle is preferable for containing the sample, as it requires less disturbance of the sample being transferred into it.

7.1.2 At Depth Soil Samples

- 1.0 Locate the sample point and clear away surface debris.
- 2.0 Use a shovel to initially open up the pile surface and then use a decontaminated bucket auger to advance the hole to the desired sampling depth.
- 3.0 Use another decontaminated bucket auger or suitable sampling device to collect the sample, and, if necessary, a decontaminated spatula or trowel to transfer the sample into the sample bottle.
- 4.0 For samples at depths greater than three feet, a hand operated hammer and extension rod may be utilized with a split spoon sampler for sample collection. Upon retrieval the split spoon is opened, its contents logged, if necessary, and the sample materials immediately transferred into a sample bottle using a decontaminated spatula, spoon, tongue depressor or trowel.

7.1.3 Sample Homogenization

- 1.0 For some types of soil samples it is desirable to thoroughly homogenize the collected sample. This can be done by using a large stainless steel mixing bowl and spoon. The sample is stirred and mixed and then placed in a sample container. The project CSAP will usually detail whether this needs to be done or not. **DO NOT HOMOGENIZE SAMPLES THAT WILL BE ANALYZED FOR VOLATILE ORGANIC COMPOUNDS.**

7.2 Sampling Equipment Decontamination

When disposable equipment is not used for sampling, the equipment must be cleaned prior to usage, after each usage (decontamination) and before usage at another sample point.

7.2.1 Inorganic Constituents

If the constituents of interest are inorganic, the following steps will be followed:

- 1) Wash with Alconox and tapwater
- 2) Rinse with tapwater
- 3) Rinse with dilute (10%) hydrochloric acid or nitric acid.
- 4) Rinse with tapwater
- 5) Rinse with distilled water or deionized water

NOTE: Dilute hydrochloric acid is generally preferred to nitric acid when cleaning stainless steel because nitric acid may oxidize stainless steel.

7.2.2 Organic Constituents

If the constituents of interest are organic, the following steps will be followed:

- 1) Wash equipment with Alconox and tapwater
- 2) Rinse with tapwater
- 3) Rinse with distilled water
- 4) Rinse with pesticide free grade isopropanol

8.0 EQUIPMENT

The materials required to perform soil pile sampling may include the following.

- Soil sampling devices:
 - Corer, tube type, 18" (Forestry Suppliers P/N 77019)
 - Trier (See Attachment 9.1)
 - Waste Pile Sampler (See Attachment 9.1)
 - Split Spoon w/hammer + extension rods (See Attachment 9.2)
 - Bucket auger, 3 1/4", with handle and ext. rods (AMS brand)
 - Scoop, plastic (VWR P/N 56920-025 or 56920-036)
 - Trowel, pointing, steel (Forestry Suppliers P/N 53717)
 - Spoon/spatula, stainless steel (VWR P/N 57952-107)
 - Tongue depressors, wood (VWR P/N 62505-006)
- Clean shovel, steel (Local Purchase)
- Sample Containers, Precleaned Glass or Plastic of appropriate size.
- Sample labels (OHM Supplied)
- Custody seals (If required for project)(OHM Supplied)
- Sample gloves, latex or nitrile (PVC may not be acceptable)
- Trash bags, 30 gallon, heavy duty (Local Purchase)
- Decontamination Supplies
 - Nitric Acid, trace metal grade (VWR P/N JT9598-0)**
 - Hydrochloric Acid, trace metal grade (VWR P/N JT9530-0)**
 - DI Water (Local Purchase)
 - Isopropyl Alcohol (2-Propanaol)(VWR P/N JT9334-3)
 - Alconox Detergent (VWR P/N 21835-032)
 - Scrub brushes and source of tap water (Local Purchase)
 - Wash buckets or tubs/pans, ~ 5-gal. size (Local Purchase)
- Field logbooks (Sample Logbook and Field Sampler's Notebook)
- 200 ft tape, Lufkin 1708D (Forestry Suppliers P/N 39961)
- Surveyors stakes (Forestry Suppliers P/N 39514)
- Marking pens, such as Sharpies and Mean Streaks
- Chain of Custody Forms (OHM Supplied)
- Compass, Silva (Type 3, 7, 20) (Forestry Suppliers or Local Purchase)
- Other OHM or site specific forms as required per CSAP
- Calculator, pocket size (Local or Individual Purchase)
- Mechanical pencil and graph paper (Local or Individual Purchase)

Specific requirements of the site sampling and analytical plan may add to or delete from the above list.

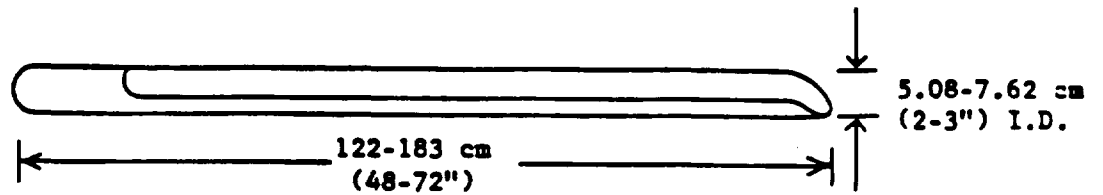
9.0 ATTACHMENTS

Figure 9.1 Trier and Waste Pile Sampler

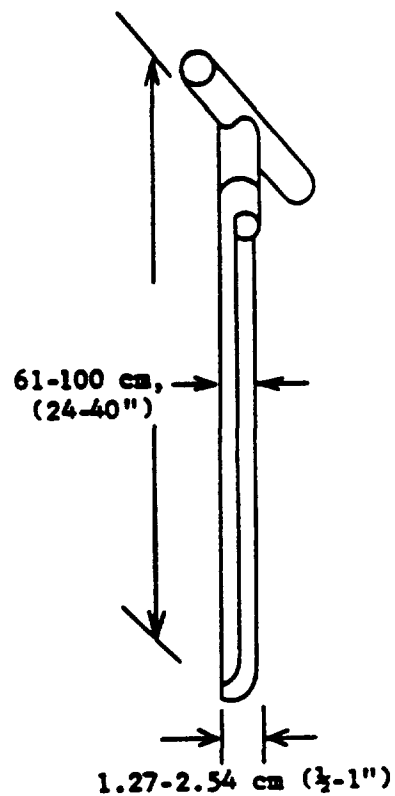
Figure 9.2 Split Spoon Sampler

Figure 9.3 Tube-type Corer and Bucket Auger

Figure 9.1 Trier and Waste Pile Sampler



Waste pile sampler.



Sampling trier.

Figure 9.2 Split Spoon Sampler

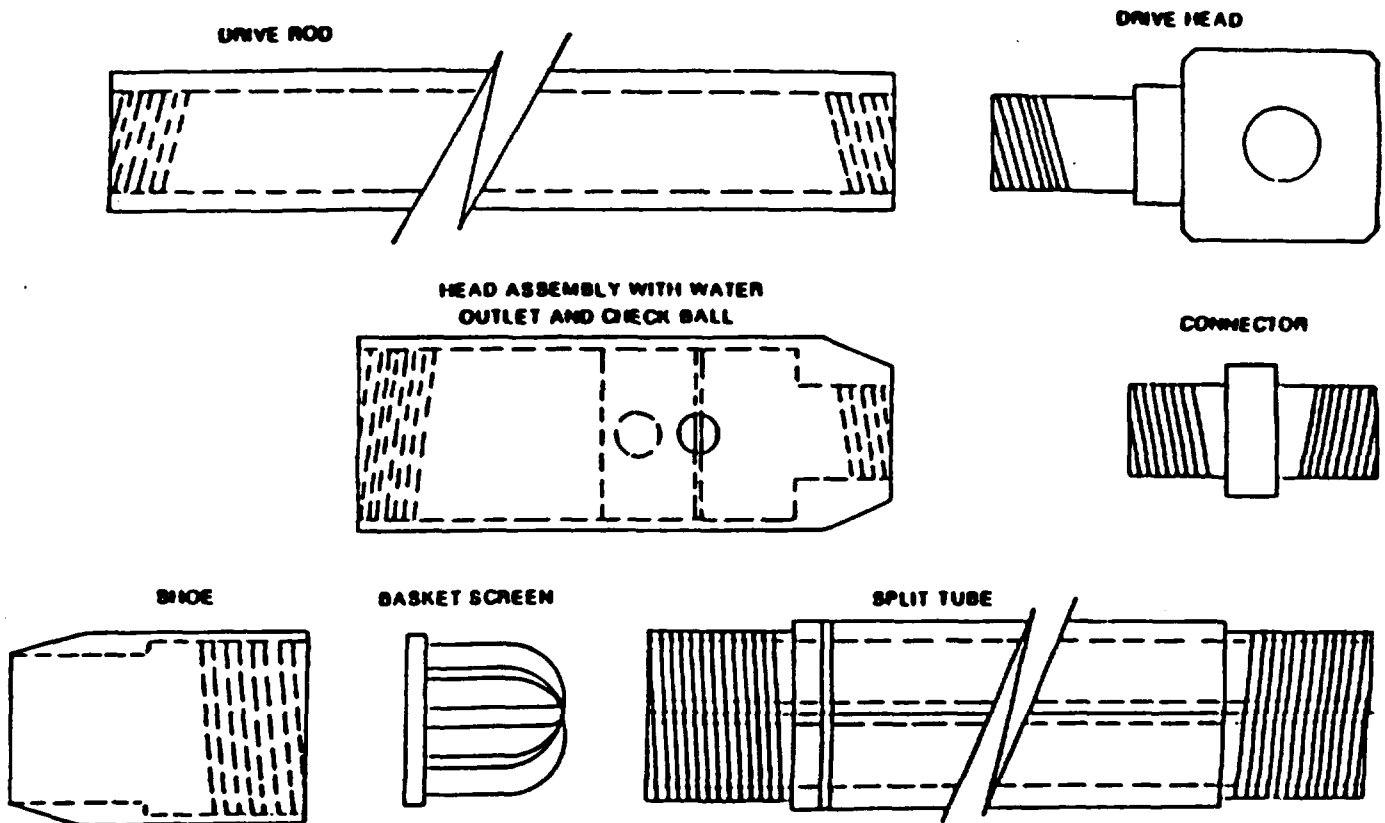


Figure 9.3 Tube-type Corer and Bucket Auger

